JACOBS ENGINEERING GROUP INC.

PLAZA OF THE AMERICAS, NORTH TOWER
700 NORTH PEARL, SUITE 300, DALLAS, TEXAS 75201
TELEPHONE (214) 969-9366

May 19, 1988

Ms. Kate Arthur
U.S. Environmental Protection Agency
Region VI
1445 Ross Avenue
Dallas, TX 75202-2733

Re: Work Assignment No.649
Submittal of Off-site Sampling Plans for Vertac Chemical Site - Jacksonville, Arkansas.

Dear Ms. Arthur:

Enclosed with this letter are three (3) copies of the final Off-site Sampling Plan for the Vertac Chemical Site as specified in the Work Assignment No.649.

If you have any questions, please do not hesitate to contact me at 969-9366.

Very truly yours,
JACOBS ENGINEERING GROUP INC.

Linda Chapman
Work Assingment Manager

GW/gl

5/19/88

ENVIRONMENTAL PROTECTION AGENCY

TECHNICAL ENFORCEMENT SUPPORT
AT
HAZARDOUS WASTE SITES

TES IV

CONTRACT NO. 68-01-7351
WORK ASSIGNMENT NO. 649
OFF-SITE SAMPLING PLAN
FOR

Vertac Chemical Site
Jacksonville, Arkansas

CERCLA

U.S. EPA Region VI

JACOBS ENGINEERING GROUP INC.
PROJECT NO.05-B649-00

DCN:TES4-JO6-00649-N1-002514

WORK ASSIGNMENT NO. 649
Sampling Plan 1

INTRODUCTION

A statistical sampling effort will be conducted in Jacksonville, Arkansas to determine the representative concentrations of TCDD in Vertac Chemical Corporation (Vertac) offsite areas where elevated levels of dioxins have been detected in grab samples, as well as in other offsite areas of potential risks. Three separate sampling plans were requested under this Work Assignment. These plans address: 1) creek sampling; 2) lake and pond sampling; and 3) residential sampling.

SITE BACKGROUND

Herbicides, including di- and tri-chlorophenoxyacetic acids, have been manufactured and formulated at the Vertac Plant in Jacksonville, Arkansas over the last 30+ years. Tetrachlorodibenzo-p-dioxin (TCDD) is a by-product of the production of these herbicides. Herbicides wastes which contained TCDD were discharged into the sanitary sewer and into Rocky Branch, a small watercourse that flows into Bayou Meto. Subsequently the downstream wastewater treatment facilities, Bayou Meto, and flood plains of Rocky Branch and Bayou Meto became contaminated with TCDD.

Attention was first focused on the Vertac site as a possible source of TCDD contamination after the National Dioxin Survey of 1978. Since then several investigations, including the RI conducted by the EPA, have confirmed TCDD contamination in the wastewater facilities (a sanitary sewer system, an old sewage treatment plant which is not in service and, an active aeration pond and two oxidation basins); in two waterways which drain this area and receive treated wastewater effluent (Rocky Branch and Bayou Meto); and in the flood plains adjacent to these waterways.

PROPOSED SAMPLE LOCATIONS AND ANALYSES

All sampling will be conducted by the Contractor or a potentially responsible party or parties (PRPs) in accordance with this sampling plan. EPA will be present in an oversight role and will split 10 percent of the samples analyzed. Approximate sample locations are shown on Map 1. This sampling plan addresses the aforementioned (1) creek sampling.

The Contractor or PRP(s) will sample:

- o Rocky Branch Creek and its tributary which border the residential area adjacent (south) of the Vertac Plant property. (Map 1-Areas A,B)
- o Outfall from the Jacksonville Sewage Treatment Plant lagoons. (Map 1-Area G3)
- o Areas of Bayou Meto which earlier sampling showed dioxin levels in excess of

0.5 ppb. (Map 1 - Areas J,K,L)

Approximately 40 soil and sediment or composite samples will be analyzed by the Contractor or PRP(s) and approximately 4 of those will be split with EPA (10 percent of the total number of samples analyzed). (See sample locations on Map 1).

All samples will be analyzed for total TCDD on a 48 hour turnaround , semi-isomer specific analysis by tandem mass spectrometry (GC/MS/MS). (Attachment 1). This allows for a fast quantitative screening of samples. Any samples with levels greater than 1.0 ppb will be reanalyzed using an isomer-specific analysis for 2,3,7,8-TCDD only.

The number and location of sample points in a given sampling plan is dependent on the number of samples necessary to meet the sampling objectives, which for this plan is that of screening. Composite sampling will be used in this sampling project. Composite sampling techniques are discussed under "Sample Methodology."

QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

The purpose of collecting split samples is to perform a quality assurance monitoring of the sampling procedures. The sampling methods detailed in this sampling plan shall be adhered to strictly and any deviations or additions to this plan will be carefully documented in a field notebook and approved by the EPA Remedial Project Manager (RPM) prior to commencement of sampling, or changes will be approved in accordance with procedures included in a Consent Administrative Order.

Quality Control (QC) samples will include duplicates, field blanks, (background) and equipment rinsate blanks (if necessary). Field blanks will consist of an appropriate matrix chosen by the RPM and should be taken offsite. These samples should also have the same soil characteristics of the sampling area. QC samples should comprise 25% of the total number of samples taken. (Attachment 3)

EPA representatives will obtain the samples from the Contractor or PRP(s). The EPA representative shall receive custody of the split samples and prepare them for shipment to a designated laboratory. All actual sampling will be conducted by the Contractor or PRP(s).

SAMPLE METHODOLOGY (GENERAL)

Specific equipment to be used in the sampling procedures should be noted in the decontamination section of the Site Safety Plan to be approved by the EPA RPM. The method used to flag the sample locations will also be determined by the EPA RPM.

Soils

Each aliquot will consist of 2 spoonfuls of soil of approximate equal weight taken from the top 3 inches of soil. Coarser fragments (or grass) should not be included in the sample. The soil should be collected in a container, such as a stainless steel

pan, which will allow for the proper mixing of the soil. It is vital that the soil be homogenized. After the soil has been well mixed, it will be transferred to a glass sample container with teflon lid.

The use of spoons or scoops (stainless steel) allows for rapid sample collection. However, a small soil corer (stainless steel) will provide consistent soil volume and depth.

Sediments

Sediment samples will be taken with a stainless steel tube, hollow auger or another RPM approved method. After the sampling apparatus is extracted from the sediment bed, an implement such as a stainless steel spoon should be used to remove the sample and to place in a glass sample jar.

In the event the creek beds are dry, then spoons, scoops, or small corers could be used.

SAMPLING METHODOLOGY (SPECIFIC)

This sampling plan addresses the creek areas previously noted. (Map 1- Areas A,B,G3,J,K,L). Sampling will be conducted in the following manner:

Soil:

- The creeks will be divided into 500 feet sections for each composite sample.
- Samples will be taken every 10 feet on the wall of the creek on each side of the creek.
- Samples will be taken 0-6" above water level; 7"-12" above water level; 13"-36" above water level. (Only the 0-6" sample will be analyzed. If levels at or above 1 ppb are present then the next level of samples will be analyzed). "Water Level" will be determined on-site by the Contractor and EPA RPM using established elevation bench marks.

An exception to this method will be made for Location G3. For the outfall area, samples will be taken approximately every 15 feet (rather than every 10 feet) for a total of 50 sample portions, one composite sample. Samples will be taken on only one side of the creek, since the waterway is straight.

Sediment: (conducted at mid-stream)

- One bottom grab sample will be taken in the west leg of Rocky Branch Creek at the southern boundary of the Vertac site, in the east leg of Rocky Branch Creek at the southern boundary of the Vertac site, and the confluent of the two legs for a total of 3 samples. Samplers should obtain enough sediment to satisfy laboratory needs. Each sample will be analyzed.
- Bottom samples will be taken approximately every 15 feet in Location G3 for a total of 50 sample portions, one composite sample.

- o Sample grids may need to be changed once on site if necessary to adjust for site specific needs. This will be determined by the EPA RPM.

FIELD DECONTAMINATION OF SAMPLING EQUIPMENT

All equipment used in the sampling event shall be decontaminated prior to initial use and between samples to prevent cross-contamination between samples. The procedure will involve washing tools with a solution of laboratory grade detergent and potable water, followed by rinses with fresh potable water, distilled water, and finally hexane. When possible, disposable equipment should be used in order to minimize decontamination time in the field. Exact procedures for decontamination and ultimate disposal of all disposed equipment will be addressed by the Contractor or PRP(s) in the site safety plan.

FIELD NOTEBOOKS AND PHOTOGRAPHS

All pertinent field survey and sampling effort information shall be recorded in a logbook during each day of field effort. A log book will be assigned to the field task and will have a unique document control number. The logbook will be bound and will have consecutively numbered pages. The field team leader will be responsible for ensuring that sufficient detail is recorded. Logbooks will contain sufficient information so that field activities can be reconstructed without relying on the memory of the field crew. All entries shall be made with indelible ink. Each day's entries will be initialed and dated at the end by the author, and a line will be drawn through the remainder of the page. All corrections shall consist of line-out deletions that are initialed. Entries in logbooks will include:

1) Date and time of starting work.

2) Name of field task leader and team members.

3) Purpose of proposed work effort.

4) Description of work area, including information on photographs taken.

5) Location of work area, including map reference.

6) Details of work effort, particularly any deviation from the field operations plan or standard operating procedures.

7) Field observations.

8) Personnel and equipment decontamination procedures.

9) Weather conditions.

For sampling efforts, specific details for each sample will be recorded on separate sample data sheets. However, in addition to the items listed above, the following general information shall be included in the logbook during sampling efforts:

1) Type and number of samples.

2) Sampling method, particularly deviations from the standard operating procedures.

3) Sample location and number.

Strict custody procedures will be maintained with the field logbooks. While being used in the field, logbooks shall remain with the field team at all times. Photocopies of the logbooks will be used as working documents.

Photographs will be taken with a 35 mm camera with a 50 mm lens. For each photograph taken, several items will be recorded in the field logbooks:

1) Date and time.

2) Name of photographer.

3) Name of site and field task.

4) General direction faced and description of the subject.

5) Location on-site.

6) Sequential number of the photograph and roll number.

Photographic information from the logbooks will be photocopied and placed in the file accompanying the slides or prints.

HEALTH AND SAFETY CONSIDERATIONS

The Contractor or PRP(s) will submit a Site Safety Plan to the EPA RPM for approval. All field personnel will have thoroughly reviewed the approved Site Safety Plan and will understand the safety considerations and should be familiar with emergency procedures prior to site entry. All field personnel at the site will have been trained and authorized to use respiratory protection, if necessary.

It will be the responsibility of the EPA RPM to coordinate with City Officials (regarding the police firing range adjacent to the old sewage treatment plant and access to the west Sewage Treatment Plant) and contact individuals (regarding access to private yards and leashing (if necessary) of dogs during sampling).

SAMPLE PACKAGING, LABELING AND PRESERVATION

The sample packaging/staging area location will be determined daily based on sample locations and safety. All samples will be transferred into glassware and capped immediately with teflon-lined lids. Bottles and caps will be supplied by an EPA approved sample bottle supplier.

Upon containerization, each sample will be labeled with a site name, sample

location, unique sample number, collection time, and type of analysis to be performed. Duplicate containers will be assigned different sample numbers. Labels are provided by the bottle supplier. The label is then covered with wide clear tape to protect the label. A serialized sample tag, provided by EPA, will be tied to the bottle neck. A chain-of-custody seal will be filled out, signed, and then afixed over the bottle cap. The bottles will then be placed in a zip-lock plastic bag. The bottles should then be placed in individual sample cans. An absorbent material should be poured into the can to cushion the glassware and absorb any spills due to breakage during transport. The can should then be closed and a completed Chain-of-Custody seal attached across the top.

All samples will be placed in coolers for overnight delivery to a designated laboratory. Additional packing material may be needed to fill voids in the cooler.

CHAIN OF CUSTODY

A required part of any sampling and analytical program is to protect the integrity of, and keep track of, samples from collection to data reporting. This includes the ability to trace the possession and handling of samples from the time of collection, through analysis and final disposition. This documentation is referred to as "chain-of-custody."

Numerous sample identification documents will be used to maintain identification and chain-of-custody of all samples collected and to control sample disposition. These include: sample identification tags, receipt for samples forms, custody seals, chain-of-custody records, sample labels, and possibly other laboratory specific forms. All forms should be filled out with waterproof ink.

All necessary sample identification tags will be provided or approved by the EPA and the serial numbers will be recorded in the field notebook. Sample tags will be attached to each sample collected. Unused tags will be returned to the Field Team Leader.

Samples shipped to the designated laboratory will be placed in coolers sealed with custody seals. Two seals will be placed on each cooler, one at the front and one at the back. Clear tape will be placed over the seals to ensure that seals are not accidentally broken during shipment. All samples will be shipped under requirements listed in 49 CFR 172.101. Any additional requirements (i.e. labels) by the delivery service should be determined prior to the commencement of sampling.

All samples will be accompanied by a Chain-of-Custody Record. When transferring samples, the individuals relinquishing and receiving should sign, date, and note the time on the record. This record will be used to document sample custody transfer from the sampler to another contractor's team member, to a shipper, or to the designated laboratory.

Samples will be packaged properly for shipment and dispatched to the designated laboratory for analysis, with a separate Chain-of-Custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information will be entered in the "remarks" section of the Chain-of-Custody Record.

All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record accompanies the shipment, and the yellow copy should be given to the Contractor's or PRP(s) Field Team Leader.

Other forms may be required, dependent upon the laboratory selected. It may also be necessary to notify the Sample Management Office (SMO) in Alexandria, Virginia of the shipment information (i.e. laboratory name, airbill number) if a Contract Laboratory Program (CLP) laboratory is being utilized. This information will be obtained from the RPM upon selection of the designated laboratory.

SAMPLING PERSONNEL/ACTIVITIES SCHEDULE

Contractor or the PRP(s) shall provide the EPA RPM with the name of the Field Team Leader and names of other team members. Contractor or the PRP(s) shall be able to provide, at EPA request, proof of OSHA approved safety certification for all team members.

Sampling activities will be scheduled according to laboratory availability, and an agreed upon time-frame between EPA and the Contractor or the PRP(s).

WORK ASSIGNMENT NO. 649
Sampling Plan 2

INTRODUCTION

A statistical sampling effort will be conducted in Jacksonville, Arkansas to determine the representative concentrations of TCDD in Vertac Chemical Corporation (Vertac) offsite areas where elevated levels of dioxins have been detected in grab samples, as well as in other offsite areas of potential risks. Three separate sampling plans were requested under this Work Assignment. These plans address: 1) creek sampling; 2) lake and pond sampling; and 3) residential sampling.

SITE BACKGROUND

Herbicides, including di- and tri-chlorophenoxyacetic acids, have been manufactured and formulated at the Vertac Plant in Jacksonville, Arkansas over the last 30+ years. Tetrachlorodibenzo-p-dioxin (TCDD) is a by-product of the production of these herbicides. Herbicides wastes which contained TCDD were discharged into the sanitary sewer and into Rocky Branch, a small watercourse that flows into Bayou Meto. Subsequently the downstream wastewater treatment facilities, Bayou Meto, and flood plains of Rocky Branch and Bayou Meto became contaminated with TCDD.

Attention was first focused on the Vertac site as a possible source of TCDD contamination after the National Dioxin Survey of 1978. Since then several investigations, including the RI conducted by the EPA, have confirmed TCDD contamination in the wastewater facilities (a sanitary sewer system, an old sewage treatment plant which is not in service and, an active aeration pond and two oxidation basins); in two waterways which drain this area and receive treated wastewater effluent (Rocky Branch and Bayou Meto); and in the flood plains adjacent to these waterways.

PROPOSED SAMPLE LOCATIONS AND ANALYSES

All sampling will be conducted by the Contractor or a potentially responsible party or parties (PRPs) in accordance with this sampling plan. EPA will be present in an oversight role and will split 10 percent of the samples analyzed. Approximate sample locations are shown on Map 1. This sampling plan addresses the aforementioned (2) lake and pond sampling.

The Contractor or PRP(s) will sample:

- o Aeration pond - Jacksonville Wastewater Treatment Plant. (Map 1 - Area F)
- o Lagoons - Jacksonville Wastewater Treatment Plant. (Map 1 - Area G1/G2)
- o Lake Dupree. (Map 1 - Area I)

Approximately 22 soil and sediment samples will be analyzed by the Contractor or PRP(s) and approximately 2 of those will be split with EPA (10 percent of the total number of samples analyzed). (See sample locations on Map 1).

All samples will be analyzed for total TCDD on a 48 hour turnaround , semi-isomer specific analysis by tandem mass spectrometry (GC/MS/MS). (Attachment 1). This allows for a fast quantitative screening of samples. Any samples with levels greater than 1.0 ppb will be reanalyzed using an isomer-specific analysis for 2,3,7,8-TCDD only.

The number and location of sample points in a given sampling plan is dependent on the number of samples necessary to meet the sampling objectives, which for this plan is that of screening. Composite sampling will be used in this sampling project. Composite sampling techniques are discussed under "Sample Methodology."

QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

The purpose of collecting split samples is to perform a quality assurance monitoring of the sampling procedures. The sampling methods detailed in this sampling plan shall be adhered to strictly and any deviations or additions to this plan will be carefully documented in a field notebook and approved by the EPA Remedial Project Manager (RPM) prior to commencement of sampling, or changes will be approved in accordance with procedures included in a Consent Administrative Order.

Quality Control (QC) samples will include duplicates, field blanks, (background) and equipment rinsate blanks (if necessary). Field blanks will consist of an appropriate matrix chosen by the RPM and should be taken offsite. These samples should also have the same soil characteristics of the sampling area. QC samples should comprise 25% of the total number of samples taken. (Attachment 3)

SAMPLE METHODOLOGY (GENERAL)

Specific equipment to be used in the sampling procedures should be noted in the decontamination section of the Site Safety Plan to be approved by the EPA RPM. The method used to flag the sample locations will also be determined by the EPA RPM.

Soils

Each aliquot will consist of 2 spoonfuls of soil of approximate equal weight taken from the top 3 inches of soil. Coarser fragments (or grass) should not be included in the sample. The soil should be collected in a container, such as a stainless steel pan, which will allow for the proper mixing of the soil. It is vital that the soil be homogenized. After the soil has been well mixed, it will be transferred to a glass sample container with teflon lid.

The use of spoons or scoops (stainless steel) allows for rapid sample collection. However, a small soil corer (stainless steel) will provide consistent soil volume and depth.

Sediments

Where noted, "top" sediment samples are to be regarded as the material on the surface of the bed on the lagoons, lakes, and aeration pond. "Bottom" sediment refers to the sediment directly above bedrock, i.e. point of resistance.

Sediment samples will be taken with a stainless steel tube, hollow auger or another RPM approved method. After the sampling apparatus is extracted from the sediment bed, an implement such as a stainless steel spoon should be used to remove the sample and to place in a glass sample jar.

In the event the creek beds are dry, then spoons, scoops, or small corers could be used.

SAMPLING METHODOLOGY (SPECIFIC)

This sampling plan addresses the lake and pond areas previously noted. (Map 1 - Area F,G1/G2;I). Sampling will be conducted in the following manner:

Area F - Aeration Pond (Jacksonville Wastewater Treatment)

Soil:

- Samples will be taken approximately every 20 feet on the dike surrounding the pond for a total of 50 sample locations, one composite sample.
- Samples will be taken within 10 feet of the dike's edge, on the water (freeboard) side.

Sediment:

- The aeration basin will be divided into 3 equal grids.
- Each grid will have 2 composite samples - 1 top sediment, 1 bottom sediment.
- Each composite sample will consist of no less than 6 aliquots.
- Aliquot locations will be selected randomly by the Contractor or PRP(s) and/or the EPA RPM and approved by the RPM.

Area G1 - Lagoon (Jacksonville Wastewater Treatment)

Soil:

- Samples will be taken approximately every 55 feet on the road/dike surrounding the lagoon on the 3 outer sides. This will be 25 sample portions and does not include the road/dike separating the 2 lagoons.
- Samples will be taken within 10 feet of the water's edge.

Sediment:

- The lagoon will be divided into 2 equal grids - east and west.
- Each grid will have 2 composite samples - 1 top sediment, 1 bottom sediment.
- Each composite sample will consist of no less than 4 aliquots.
- Aliquot locations will be selected randomly by the Contractor or PRP(s) and/or the EPA RPM and approved by the RPM.

Area G2 - Lagoon (Jacksonville Wastewater Treatment)

This lagoon will be sampled in the same manner as Area G1.

Area I - Lake Dupree

Soil:

- Samples will be taken approximately every 60 feet on the dike surrounding the lake for a total of 50 sample portions, one composite sample.
- Samples will be taken within 5 feet of the water's edge.

Sediment:

- The lake will be divided into 4 equal grids.
- Each grid will have 1 composite sample - the top sediment.
- Each composite will consist of no less than 6 aliquots.
- Aliquot locations will be selected randomly by the Contractor or PRP(s) and/or the EPA RPM and approved by the RPM.

FIELD DECONTAMINATION OF SAMPLING EQUIPMENT

All equipment used in the sampling event shall be decontaminated prior to initial use and between samples to prevent cross-contamination between samples. The procedure will involve washing tools with a solution of laboratory grade detergent and potable water, followed by rinses with fresh potable water, distilled water, and

finally hexane. When possible, disposable equipment should be used in order to minimize decontamination time in the field. Exact procedures for decontamination and ultimate disposal of all disposed equipment will be addressed by the Contractor or PRP(s) in the site safety plan.

FIELD NOTEBOOKS AND PHOTOGRAPHS

All pertinent field survey and sampling effort information shall be recorded in a logbook during each day of field effort. A log book will be assigned to the field task and will have a unique document control number. The logbook will be bound and will have consecutively numbered pages. The field team leader will be responsible for ensuring that sufficient detail is recorded. Logbooks will contain sufficient information so that field activities can be reconstructed without relying on the memory of the field crew. All entries shall be made with indelible ink. Each day's entries will be initialed and dated at the end by the author, and a line will be drawn through the remainder of the page. All corrections shall consist of line-out deletions that are initialed. Entries in logbooks will include:

1) Date and time of starting work.

2) Name of field task leader and team members.

3) Purpose of proposed work effort.

4) Description of work area, including information on photographs taken.

5) Location of work area, including map reference.

6) Details of work effort, particularly any deviation from the field operations plan or standard operating procedures.

7) Field observations.

8) Personnel and equipment decontamination procedures.

9) Weather conditions.

For sampling efforts, specific details for each sample will be recorded on separate sample data sheets. However, in addition to the items listed above, the following general information shall be included in the logbook during sampling efforts:

1) Type and number of samples.

2) Sampling method, particularly deviations from the standard operating procedures.

3) Sample location and number.

Strict custody procedures will be maintained with the field logbooks. While being used in the field, logbooks shall remain with the field team at all times. Photocopies of the logbooks will be used as working documents.

Photographs will be taken with a 35 mm camera with a 50 mm lens. For each photograph taken, several items will be recorded in the field logbooks:

1) Date and time.

2) Name of photographer.

3) Name of site and field task.

4) General direction faced and description of the subject.

5) Location on-site.

6) Sequential number of the photograph and roll number.

Photographic information from the logbooks will be photocopied and placed in the file accompanying the slides or prints.

HEALTH AND SAFETY CONSIDERATIONS

The Contractor or PRP(s) will submit a Site Safety Plan to the EPA RPM for approval. All field personnel will have thoroughly reviewed the approved Site Safety Plan and will understand the safety considerations and should be familiar with emergency procedures prior to site entry. All field personnel at the site will have been trained and authorized to use respiratory protection, if necessary.

It will be the responsibility of the EPA RPM to coordinate with City Officials (regarding the police firing range adjacent to the old sewage treatment plant and access to the west Sewage Treatment Plant) and contact individuals (regarding access to private yards and leashing (if necessary) of dogs during sampling).

SAMPLE PACKAGING, LABELING AND PRESERVATION

The sample packaging/staging area location will be determined daily based on sample locations and safety. All samples will be transferred into glassware and capped immediately with teflon-lined lids. Bottles and caps will be supplied by an EPA approved sample bottle supplier.

Upon containerization, each sample will be labeled with a site name, sample location, unique sample number, collection time, and type of analysis to be performed. Duplicate containers will be assigned different sample numbers. Labels are provided by the bottle supplier. The label is then covered with wide clear tape to protect the label. A serialized sample tag, provided by EPA, will be tied to the bottle neck. A chain-of-custody seal will be filled out, signed, and then afixed over the bottle cap. The bottles will then be placed in a zip-lock plastic bag. The bottles should then be placed in individual sample cans. An absorbent material should be poured into the can to cushion the glassware and absorb any spills due to breakage during transport. The can should then be closed and a completed Chain-of-Custody seal attached across the top.

All samples will be placed in coolers for overnight delivery to a designated laboratory. Additional packing material may be needed to fill voids in the cooler.

CHAIN OF CUSTODY

A required part of any sampling and analytical program is to protect the integrity of, and keep track of, samples from collection to data reporting. This includes the ability to trace the possession and handling of samples from the time of collection, through analysis and final disposition. This documentation is referred to as "chain-of-custody."

Numerous sample identification documents will be used to maintain identification and chain-of-custody of all samples collected and to control sample disposition. These include: sample identification tags, receipt for samples forms, custody seals, chain-of-custody records, sample labels, and possibly other laboratory specific forms. All forms should be filled out with waterproof ink.

All necessary sample identification tags will be provided or approved by the EPA and the serial numbers will be recorded in the field notebook. Sample tags will be attached to each sample collected. Unused tags will be returned to the Field Team Leader.

Samples shipped to the designated laboratory will be placed in coolers sealed with custody seals. Two seals will be placed on each cooler, one at the front and one at the back. Clear tape will be placed over the seals to ensure that seals are not accidentally broken during shipment. All samples will be shipped under requirements listed in 49 CFR 172.101. Any additional requirements (i.e. labels) by the delivery service should be determined prior to the commencement of sampling.

All samples will be accompanied by a Chain-of-Custody Record. When transferring samples, the individuals relinquishing and receiving should sign, date, and note the time on the record. This record will be used to document sample custody transfer from the sampler to another contractor's team member, to a shipper, or to the designated laboratory.

Samples will be packaged properly for shipment and dispatched to the designated laboratory for analysis, with a separate Chain-of-Custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information will be entered in the "remarks" section of the Chain-of-Custody Record.

All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record accompanies the shipment, and the yellow copy should be given to the Contractor's or PRP(s) Field Team Leader.

Other forms may be required, dependent upon the laboratory selected. It may also be necessary to notify the Sample Management Office (SMO) in Alexandria, Virginia of the shipment information (i.e. laboratory name, airbill number) if a Contract Laboratory Program (CLP) laboratory is being utilized. This information will be obtained from the RPM upon selection of the designated laboratory.

SAMPLING PERSONNEL/ACTIVITIES SCHEDULE

Contractor or the PRP(s) shall provide the EPA RPM with the name of the Field Team Leader and names of other team members. Contractor or the PRP(s) shall be able to provide, at EPA request, proof of OSHA approved safety certification for all team members.

Sampling activities will be scheduled according to laboratory availability, and an agreed upon time-frame between EPA and the Contractor or the PRP(s).

WORK ASSIGNMENT NO. 649
Sampling Plan 3

INTRODUCTION

A statistical sampling effort will be conducted in Jacksonville, Arkansas to determine the representative concentrations of TCDD in Vertac Chemical Corporation (Vertac) offsite areas where elevated levels of dioxins have been detected in grab samples, as well as in other offsite areas of potential risks. Three separate sampling plans were requested under this Work Assignment. These plans address: 1) creek sampling; 2) lake and pond sampling; and 3) residential sampling.

SITE BACKGROUND

Herbicides, including di- and tri-chlorophenoxyacetic acids, have been manufactured and formulated at the Vertac Plant in Jacksonville, Arkansas over the last 30+ years. Tetrachlorodibenzo-p-dioxin (TCDD) is a by-product of the production of these herbicides. Herbicides wastes which contained TCDD were discharged into the sanitary sewer and into Rocky Branch, a small watercourse that flows into Bayou Meto. Subsequently the downstream wastewater treatment facilities, Bayou Meto, and flood plains of Rocky Branch and Bayou Meto became contaminated with TCDD.

Attention was first focused on the Vertac site as a possible source of TCDD contamination after the National Dioxin Survey of 1978. Since then several investigations, including the RI conducted by the EPA, have confirmed TCDD contamination in the wastewater facilities (a sanitary sewer system, an old sewage treatment plant which is not in service and, an active aeration pond and two oxidation basins); in two waterways which drain this area and receive treated wastewater effluent (Rocky Branch and Bayou Meto); and in the flood plains adjacent to these waterways.

PROPOSED SAMPLE LOCATIONS AND ANALYSES

All sampling will be conducted by the Contractor or a potentially responsible party or parties (PRPs) in accordance with this sampling plan. EPA will be present in an oversight role and will split 10 percent of the samples analyzed. Approximate sample locations are shown on Map 1. This sampling plan addresses the aforementioned (3) residential sampling.

The Contractor or PRP(s) will sample:

- Specific areas of the residential area south of the Vertac property. (Map 1 - Areas C,D).
- Areas of the residential area south of the Vertac property - gross (large) grid. (Map 1 - Area D)

- o The Old Sewage Treatment Plant. (Map 1 - Area E1/E2)
- o Ditch containing manhole 2043 near Redmond Road and Hwy. 67. (Map 1 - Area H)
- o Dry stream bed parallel to Rocky Branch, south of Redmond Road. (Map 1 - Area M)

Approximately 75 soil and sediment or composite samples will be analyzed by the Contractor or PRP(s) and approximately 7 of those will be split with EPA (10 percent of the total number of samples analyzed). (See sample locations on Map 1).

All samples will be analyzed for total TCDD on a 48 hour turnaround , semi-isomer specific analysis by tandem mass spectrometry (GC/MS/MS). (Attachment 1). This allows for a fast quantitative screening of samples. Any samples with levels greater than 1.0 ppb will be reanalyzed using an isomer-specific analysis for 2,3,7,8-TCDD only.

The number and location of sample points in a given sampling plan is dependent on the number of samples necessary to meet the sampling objectives, which for this plan is that of screening. Composite sampling will be used in this sampling project. Composite sampling techniques are discussed under "Sample Methodology."

This plan is based on random statistical sampling procedures to ensure a 95% confidence factor (Attachment 2).

QUALITY ASSURANCE/QUALITY CONTROL (QA/QC) PROCEDURES

The purpose of collecting split samples is to perform a quality assurance monitoring of the sampling procedures. The sampling methods detailed in this sampling plan shall be adhered to strictly and any deviations or additions to this plan will be carefully documented in a field notebook and approved by the EPA Remedial Project Manager (RPM) prior to commencement of sampling, or changes will be approved in accordance with procedures included in a Consent Administrative Order.

Quality Control (QC) samples will include duplicates, field blanks, (background) and equipment rinsate blanks (if necessary). Field blanks will consist of an appropriate matrix chosen by the RPM and should be taken offsite. These samples should also have the same soil characteristics of the sampling area. QC samples should comprise 25% of the total number of samples taken. (Attachment 3)

SAMPLE METHODOLOGY (GENERAL)

Specific equipment to be used in the sampling procedures should be noted in the decontamination section of the Site Safety Plan to be approved by the EPA RPM. The method used to flag the sample locations will also be determined by the EPA RPM.

Soils

Each sample will consist of 2 spoonfuls of soil of approximate equal weight taken from the top 3 inches of soil. Coarser fragments (or grass) should not be included in the sample. The soil should be collected in a container, such as a stainless steel pan, which will allow for the proper mixing of the soil. It is vital that the soil be homogenized. After the soil has been well mixed, it will be transferred to a glass sample container with teflon lid.

The use of spoons or scoops (stainless steel) allows for rapid sample collection. However, a small soil corer (stainless steel) will provide consistent soil volume and depth.

SAMPLING METHODOLOGY (SPECIFIC)

This sampling plan addresses the residential areas as previously noted. Fine grid sampling methods will be used at Location C. A variation of fine grid sampling will be used at Locations E1/E2, H, and M and random grab sampling will be used at Location D.

Fine grid sampling will be based on the document "A Sampling Strategy for Cleanups of Dioxins in Soils" (Attachment 2). Modifications will be made as deemed appropriate by the EPA RPM.

The maximum area for one grid will be 5,000 sq. ft. The grid will be subdivided into areas no larger than 10 ft x 10 ft. Each sub grid will be marked in the center. Two spoonfuls of soils will be taken approximately 2.5 ft to the north of the mark, 2.5 ft to the west of the mark, and 2.5 ft to the south of the mark. All north aliquots will be composited to form one sample, all west another, and all south the third. All composite samples will be analyzed by the rapid determination screening method.

Samples will also be taken to confirm mixing technique. The number and type of samples will be determined by the EPA RPM.

The sampling method used for each location (Figures C,D,E,H,M) is as follows:

Area C - Residential

- This area will be gridded according to fine grid procedures.
- Grids will begin at the Rocky Branch/tributary confluent and go northward to the Vertac fence line.
- Grids will be adjacent to Rocky Branch and its tributary, on the residential side of the creek.
- Grids will be approximately 20'x 250' in the wooded area south of 2111 West Lane on the west leg of Rocky Branch Creek and south of the apartments on the east leg. Grids 20' x property lines shall be used in all other appropriate areas. Two adjacent grids each 20' in width will be sampled.

- Any natural occurrences such as drainage areas should be considered when sampling.

- If sample results are above 1 ppb for the grid closest to the creek, then the composite samples from the adjacent grid (further into the residential area) will be analyzed.

- The manhole area (manhole No. 2734) in the side yard at 612 Oakley will be fine gridded. Grid size and direction will be determined at the location dependent on site-specific needs.

- The manhole area (manhole No. 2745) in the backyard at 1704 Hill Street will be fine gridded. Grid size and direction will be determined at the location dependent on site-specific needs.

Area D - Residential

- This area will be divided into 2 large grids - east and west.

- Three samples will be taken in each grid.

- Samples will be random grab samples, not composites. Locations will be determined by the Contractor or PRP(s) and the EPA RPM.

Area E1/E2 - Old Sewage Treatment Plant

Drying Bed Area:

- This area will have 2 grids - drying beds and non-bed areas.

- A total of 50 samples will be taken in the sludge beds for one composite sample. Locations will be determined at the time of sampling by the EPA RPM.

- A total of 50 portions will be taken in the area outside the drying beds for one composite sample. Locations will be determined at the time of sampling by the EPA RPM.

Clarifier Area:

- This area will be considered one grid area.

- A total of 50 portions will be taken in this area for one composite sample. Locations will be determined at the time of sampling by the EPA RPM.

Area H - Drainage ditch with Manhole 2043

- The lowest level of the ditch will be considered one grid.

- The grid will be a rectangular area parallel to South Redmond Road which encompasses the area shown as Area H.

- Approximately 40 portions will be taken in the grid for one composite sample.

These samples will be taken by fine grid procedures.

Area M - Dry stream bed

- The dry stream bed will be divided into 3 grid areas - 2 equal grids south of South Redmond Road south to the drainage line from the old sewage treatment plant and 1 grid (size of each of the other grids) south of the drainage line.
- The grids will be approximately 10'x500'.
- Approximately 50 portions will be taken in each grid for one composite sample per grid.
- Samples will be taken every 10 feet at the mid-point of the creek bed, to obtain one composite sample of 50 portions.

FIELD DECONTAMINATION OF SAMPLING EQUIPMENT

All equipment used in the sampling event shall be decontaminated prior to initial use and between samples to prevent cross-contamination between samples. The procedure will involve washing tools with a solution of laboratory grade detergent and potable water, followed by rinses with fresh potable water, distilled water, and finally hexane. When possible, disposable equipment should be used in order to minimize decontamination time in the field. Exact procedures for decontamination and ultimate disposal of all disposed equipment will be addressed by the Contractor or PRP(s) in the site safety plan.

FIELD NOTEBOOKS AND PHOTOGRAPHS

All pertinent field survey and sampling effort information shall be recorded in a logbook during each day of field effort. A log book will be assigned to the field task and will have a unique document control number. The logbook will be bound and will have consecutively numbered pages. The field team leader will be responsible for ensuring that sufficient detail is recorded. Logbooks will contain sufficient information so that field activities can be reconstructed without relying on the memory of the field crew. All entries shall be made with indelible ink. Each day's entries will be initialed and dated at the end by the author, and a line will be drawn through the remainder of the page. All corrections shall consist of line-out deletions that are initialed. Entries in logbooks will include:

1) Date and time of starting work.
2) Name of field task leader and team members.
3) Purpose of proposed work effort.
4) Description of work area, including information on photographs taken.
5) Location of work area, including map reference.

6) Details of work effort, particularly any deviation from the field operations plan or standard operating procedures.

7) Field observations.

8) Personnel and equipment decontamination procedures.

9) Weather conditions.

For sampling efforts, specific details for each sample will be recorded on separate sample data sheets. However, in addition to the items listed above, the following general information shall be included in the logbook during sampling efforts:

1) Type and number of samples.

2) Sampling method, particularly deviations from the standard operating procedures.

3) Sample location and number.

Strict custody procedures will be maintained with the field logbooks. While being used in the field, logbooks shall remain with the field team at all times. Photocopies of the logbooks will be used as working documents.

Photographs will be taken with a 35 mm camera with a 50 mm lens. For each photograph taken, several items will be recorded in the field logbooks:

1) Date and time.

2) Name of photographer.

3) Name of site and field task.

4) General direction faced and description of the subject.

5) Location on-site.

6) Sequential number of the photograph and roll number.

Photographic information from the logbooks will be photocopied and placed in the file accompanying the slides or prints.

HEALTH AND SAFETY CONSIDERATIONS

The Contractor or PRP(s) will submit a Site Safety Plan to the EPA RPM for approval. All field personnel will have thoroughly reviewed the approved Site Safety Plan and will understand the safety considerations and should be familiar with emergency procedures prior to site entry. All field personnel at the site will have been trained and authorized to use respiratory protection, if necessary.

It will be the responsibility of the EPA RPM to coordinate with City Officials

(regarding the police firing range adjacent to the old sewage treatment plant and access to the west Sewage Treatment Plant) and contact individuals (regarding access to private yards and leashing (if necessary) of dogs during sampling).

SAMPLE PACKAGING, LABELING AND PRESERVATION

The sample packaging/staging area location will be determined daily based on sample locations and safety. All samples will be transferred into glassware and capped immediately with teflon-lined lids. Bottles and caps will be supplied by an EPA approved sample bottle supplier.

Upon containerization, each sample will be labeled with a site name, sample location, unique sample number, collection time, and type of analysis to be performed. Duplicate containers will be assigned different sample numbers. Labels are provided by the bottle supplier. The label is then covered with wide clear tape to protect the label. A serialized sample tag, provided by EPA, will be tied to the bottle neck. A chain-of-custody seal will be filled out, signed, and then affixed over the bottle cap. The bottles will then be placed in a zip-lock plastic bag. The bottles should then be placed in individual sample cans. An absorbent material should be poured into the can to cushion the glassware and absorb any spills due to breakage during transport. The can should then be closed and a completed Chain-of-Custody seal attached across the top.

All samples will be placed in coolers for overnight delivery to a designated laboratory. Additional packing material may be needed to fill voids in the cooler.

CHAIN OF CUSTODY

A required part of any sampling and analytical program is to protect the integrity of, and keep track of, samples from collection to data reporting. This includes the ability to trace the possession and handling of samples from the time of collection, through analysis and final disposition. This documentation is referred to as "chain-of-custody."

Numerous sample identification documents will be used to maintain identification and chain-of-custody of all samples collected and to control sample disposition. These include: sample identification tags, receipt for samples forms, custody seals, chain-of-custody records, sample labels, and possibly other laboratory specific forms. All forms should be filled out with waterproof ink.

All necessary sample identification tags will be provided or approved by the EPA and the serial numbers will be recorded in the field notebook. Sample tags will be attached to each sample collected. Unused tags will be returned to the Field Team Leader.

Samples shipped to the designated laboratory will be placed in coolers sealed with custody seals. Two seals will be placed on each cooler, one at the front and one at the back. Clear tape will be placed over the seals to ensure that seals are not accidentally broken during shipment. All samples will be shipped under requirements listed in 49 CFR 172.101. Any additional requirements (i.e. labels) by

the delivery service should be determined prior to the commencement of sampling.

All samples will be accompanied by a Chain-of-Custody Record. When transferring samples, the individuals relinquishing and receiving should sign, date, and note the time on the record. This record will be used to document sample custody transfer from the sampler to another contractor's team member, to a shipper, or to the designated laboratory.

Samples will be packaged properly for shipment and dispatched to the designated laboratory for analysis, with a separate Chain-of-Custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information will be entered in the "remarks" section of the Chain-of-Custody Record.

All shipments will be accompanied by the Chain-of-Custody Record identifying its contents. The original record accompanies the shipment, and the yellow copy should be given to the Contractor's or PRP(s) Field Team Leader.

Other forms may be required, dependent upon the laboratory selected. It may also be necessary to notify the Sample Management Office (SMO) in Alexandria, Virginia of the shipment information (i.e. laboratory name, airbill number) if a Contract Laboratory Program (CLP) laboratory is being utilized. This information will be obtained from the RPM upon selection of the designated laboratory.

SAMPLING PERSONNEL/ACTIVITIES SCHEDULE

Contractor or the PRP(s) shall provide the EPA RPM with the name of the Field Team Leader and names of other team members. Contractor or the PRP(s) shall be able to provide, at EPA request, proof of OSHA approved safety certification for all team members.

Sampling activities will be scheduled according to laboratory availability, and an agreed upon time-frame between EPA and the Contractor or the PRP(s).

RAPID DETERMINATION OF TCDD IN SOIL AND SEDIMENT USING GAS CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY

MARCH 1986

U. S. ENVIRONMENTAL PROTECTION AGENCY

REGION VII

ATTACHMENT 1

TABLE OF CONTENTS

This method is for use in the rapid determination of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) in soil and sediment, when 2,3,7,8-TCDD is known to be the principal or only tetrachlorodibenzodioxin isomer present. The method is not specific for the 2,3,7,8-TCDD isomer, unless a capillary column which separates that isomer from the other 21 TCDD isomers is employed. The method is applicable in the concentration range of 0.3-25 ug/kg.

The method employs a tandem quadrupole mass spectrometer (MS/MS) as the final detector. The specificity of detection inherent in such a system significantly reduces the need for sample cleanup. This, in turn, improves productivity and cost-effectiveness relative to other high resolution and low resolution GC/MS analysis techniques. The apparatus and methods described are designed for use in a mobile laboratory, which permits on-site analyses.

The method is intended to be used when analytical results are required rapidly, such as when site cleanup operations are in progress. Since the method is not isomer specific, false positives, including isomers other than 2,3,7,8-TCDD, may occur. But errors in this regard would be on the side of safety. Emphasis in the method is placed on avoiding false negatives, as this is a more critical consideration when public health is to be protected.

This method is restricted to use only by or under the supervision of analysts experienced in the use of gas chromatography/triple quadrupole mass spectrometers and skilled in the interpretation of mass spectra.

Because of the extreme toxicity of this compound, the analyst must prevent exposure to himself, or to others, by materials known or believed to contain 2,3,7,8-TCDD. Section IV of this method contains guidelines and protocols that serve as minimum safe-handling standards in a limited access laboratory.

Analyte	CAS Number
2,3,7,8-TCDD	1746-01-6

II. SUMMARY OF METHOD

Five (5) grams of anhydrous sodium sulfate is placed in a 10 ml serum vial and the vial with cap and septum is weighed. Approximately 5 grams of a soil sample is added and the vial is re-weighed. The sample is spiked with internal and surrogate standards of isotopically labelled 2,3,7,8-TCDD. The sample is mixed by shaking, and extracted with acetonitrile/dichloromethane in the closed vial. An aliquot of the extract is taken and, after separation from acetonitrile, the dichloromethane is used directly for GC/MS/MS analysis. Clean-up should usually not be necessary, but a clean-up procedure is included for those samples which do not meet quality assurance criteria. Concentration of the extract may be done to lower the minimum detectable concentration. Capillary column GC/MS/MS conditions are described which allow for separation of TCDD from the bulk sample matrix and measurement of TCDD in the extract.

Quantification is based on the response of native TCDD relative to the isotopically labelled TCDD internal standard. Performance is assessed based on the results for surrogate standard recoveries, EPA performance evaluation samples, spike recovery tests, and method and field blanks.

III. INTERFERENCES

Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the ions monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks as described in Section VIII.

The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the sample. 2,3,7,8-TCDD is often associated with other interfering chlorinated compounds which are at concentrations several magnitudes higher than that of 2,3,7,8-TCDD.

The use of a triple quadrupole mass spectrometer as the detector serves to minimize the influence of many of these interferents.

IV. SAFETY

The following safety practices are excerpted directly from EPA Method 613, Section 4 (July 1982 version): See following page.

treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are identified[8-10]. Benzene and 2,3,7,8-TCDD have been identified as suspected human or mammalian carcinogens.

4.2 Each laboratory must develop a strict safety program for handling of 2,3,7,8-TCDD. The following laboratory practices are recommended:

4.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.

4.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

4.2.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light with wavelength greater than 290 nm for several days. (Use F40BL lamps or equivalent.) Analyze liquid wastes and dispose of the solutions when 2,3,7,8-TCDD can no longer be detected.

4.3 Dow Chemical U.S.A. has issued the following precautions (revised 11/78) for safe handling of 2,3,7,8-TCDD in the laboratory:

4.3.1 The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Inquiries about specific operations or uses may be addressed to the Dow Chemical Company. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. 2,3,7,8-TCDD is extremely toxic to laboratory animals, but it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8-TCDD.

4.3.1.1 Protective Equipment: Throw-away plastic gloves, apron or lab coat, safety glasses and lab hood adequate for radioactive work.

4.3.1.2 Training: Workers must be trained in the proper method of removing of contaminated gloves and clothing without contacting the exterior surfaces.

4.3.1.3 Personal Hygiene: Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

4.3.1.4 Confinement: Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

4.3.1.5 Waste: Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors must be trained in safe handling of waste.

4.3.1.6 Disposal of Wastes: 2,3,7,8-TCDD decomposes above 800 °C. Low-level waste such as the absorbent paper, tissues, animal remains and plastic gloves may be burned in a good incinerator. Gross quantities (milligrams) should be packaged securely and disposed through commercial or governmental channels which are capable of handling high-level radioactive wastes or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container. Residues may then be handled as above.

4.3.1.7 Decontamination: Personal—any mild soap with plenty of scrubbing action. Glassware, Tools, and Surfaces—Chlorothene NU Solvent (Trademark of the Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished by rinsing with Chlorothene, then washing with any detergent and water. Dish water may be disposed to the sewer. It is prudent to minimize solvent wastes because they may require special disposal through commercial sources which are expensive.

4.3.1.8 Laundry: Clothing known to be contaminated should be disposed with the precautions described under "Disposal of Wastes." Lab coats or other clothing worn in 2,3,7,8-TCDD should be collected in plastic bags. Persons who convey the bags and later [illegible] the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing.

4.3.1.9 Wipe Tests: A useful method of determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper. Extraction and analysis by gas chromatography can achieve a limit of sensitivity of 0.1 μg per wipe. Less than 1 μg 2,3,7,8-TCDD per sample indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 μg on a wipe sample indicates an acute hazard and requires prompt cleaning before further use of the equipment or work space and indicates further that unacceptable work practices have been employed in the past.

4.3.1.10 Inhalation: Any procedure that may produce airborne contamination must be done with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in case of an accident.

4.3.1.11 Accidents: Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

V. APPARATUS AND MATEF LS

All glassware is initially cleaned with aqueous detergent and then rinsed with tap water, deionized water, acetone, toluene and methylene chloride. Other cleaning procedures may be used as long as acceptable method blanks are obtained.

Electronic balance, capable of weighing at least 50 g, with an accuracy of at least ± 0.05 g.

Shaker, vortex-type or equivalent

Centrifuge, 4000 rpm, capable of handling 25 mm diameter vials

Centrifuge tubes

10 ml serum vials; with teflon faced septa and aluminum caps (Chrompak 10204 and 10213 or equivalent)

1 ml serum vials; with teflon faced septa and aluminum caps (Chrompak 10201 and 10211 or equivalent)

Crimper for 10 ml serum vial (Chrompak 10233 or equivalent)

Crimper for 1 ml serum vial (Chrompak 10231 or equivalent)

Disposable teflon 0.45 micron filters (Millipore SLHV025 HB, or equivalent)

5 ml disposable Glaspak syringes (Sargent Welch S-79401-B or equivalent)

18 gauge disposable syringe needle (Sargent Welch S-79402-G or equivalent)

Disposable pipets, 5 3/4 inches x 7 mm o.d.

Glass wool, silanized

Nitrogen blowdown apparatus

Gas chromatograph - an analytical system with all required accessories including syringes and analytical columns. The injection port must be designed for capillary columns and splitless injection.

Triple quadrupole mass spectrometer with GC transfer line and glow discharge ion source (TAGA® 6000, SCIEX®, Thornhill, Ontario, Canada)

Compressed Gases:	Zero Grade Air (from distillation, not water hydrolysis) Ultra High Purity Nitrogen Ultra High Purity Argon
Column:	15 m long, wide bore fused silica capillary (eg. 0.32 mm I.D.) DB-5 1.0 micron film thickness.

VI. REAGENTS

Stock Standard Solutions

Stock standard solutions correspond to three toluene solutions containing unlabelled 2,3,7,8-TCDD at varying concentrations, and $^{13}C_{12}$-2,3,7,8-TCDD (internal standard, CASRN 80494-19-5) at a constant concentration. These solutions also contain $^{37}Cl_4$-2,3,7,8-TCDD (surrogate compound, CASRN 85508-50-5) at varying concentrations. These stock solutions are to be used in preparing the calibration standard solutions, and are to be obtained from the Quality Assurance Division, USEPA, Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. If not available from EMSL-LV, stock standard solutions may be prepared from commercially available standards. However, the accuracy of these solutions must be checked against EPA supplied standard solutions.

The three stock solutions will have the following concentrations of unlabelled, internal and surrogate standards.

Stock Solution #1 (CC1)

Unlabeled 2,3,7,8-TCDD - 0.2 ng/ul
$^{13}C_{12}$-2,3,7,8-TCDD - 1.0 ng/ul
$^{37}Cl_4$-2,3,7,8-TCDD - 0.06 ng/ul

Stock Solution #2 (CC2)

Unlabeled 2,3,7,8-TCDD - 1.0 ng/ul
$^{13}C_{12}$-2,3,7,8-TCDD - 1.0 ng/ul
$^{37}Cl_4$-2,3,7,8-TCDD - 0.12 ng/ul

Stock Solution #3 (CC3)

Unlabeled 2,3,7,8-TCDD - 5.0 ng/ul
$^{13}C_{12}$-2,3,7,8-TCDD - 1.0 ng/ul
$^{37}Cl_4$-2,3,7,8-TCDD - 0.2 ng/ul

NOTE: Store stock solutions in 1 ml amber mini-vials under refrigeration.

Calibration Standard Solutions

Calibration standard solutions are prepared to simulate the conditions of sample analysis as nearly as possible. Three calibration standard solutions are prepared from the stock standard solutions so as to contain constant amounts of internal standard (5 ug/kg equivalent) with variable amounts of unlabeled standard (1,5, and 25 ug/kg equivalent) and surrogate standard (0.3, 0.6, and 1.0 ug/kg equivalent). The equivalent concentrations are based on the use of 5-gram samples, extraction with 5 ml of 2:1 acetonitrile: dichloromethane, and a final extract volume of approximately 1.66 ml dichloromethane after removal of acetonitrile, as called for in the procedure.

Low Level

Add 750 ul of stock solution #1 to a 5 ml volumetric flask and bring to volume with dichloromethane. Mix well. This solution contains an equivalent concentration of 1 ug/kg of 2,3,7,8-TCDD, 5 ug/kg of $^{13}C_{12}$-2,3,7,8-TCDD, and 0.3 ug/kg of $^{37}Cl_4$-2,3,7,8-TCDD.

Medium Level

Add 750 ul of stock solution #2 to a 5 ml volumetric flask and bring to volume with dichloromethane. Mix well. This solution contains an equivalent concentration of 5 ug/kg of 2,3,7,8-TCDD, 5 ug/kg of $^{13}C_{12}$-2,3,7,8-TCDD, and 0.6 ug/kg of $^{37}Cl_4$-2,3,7,8-TCDD.

High Level

Add 750 ul of stock solution #3 to a 5 ml volumetric flask and bring to volume with dichloromethane. Mix well. This solution contains an equivalent concentration of 25 ug/kg of 2,3,7,8-TCDD, 5 ug/kg of $^{13}C_{12}$-2,3,7,8-TCDD, and 1.0 ug/kg of $^{37}Cl_4$-2,3,7,8-TCDD.

NOTE 1: Although the surrogate, $^{37}Cl_4$-2,3,7,8-TCDD, is present in all three level calibration solutions, only the high level solution is used for calculating the relative response factor for the surrogate.

NOTE 2: All calibration standard solutions must be stored in an isolated refrigerator and protected from light. Check these standard solutions frequently for signs of evaporation.

Sample Spiking Solution

The sample spiking solution is also to be obtained from the Quality Assurance Division, U. S. EPA Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. The spiking solution will have the following concentrations of internal and surrogate standards.

$^{13}C_{12}$-2,3,7,8-TCDD - 0.5 ng/ul
$^{37}Cl_4$-2,3,7,8-TCDD - 0.1 ng/ul

When 50 ul of this solution is spiked in 5 g of soil, the resulting concentrations in the soil are 5 ug/kg and 1 ug/kg of internal and surrogate standard, respectively.

It is recommended that approximately 2.5-5 ml of the spiking solution be transferred to a 5 ml serum vial and sealed with a septum and cap prior to each day's work for use in spiking samples that day.

NOTE: It is very important that no evaporation of sample spiking solution be allowed to occur, since the accuracy of results are directly dependent on the addition of a known amount of internal standard.

Field Blank Spiking Solution

The field blank spiking solution is also to be obtained from the Quality Assurance Division, U. S. EPA, Environmental Monitoring Systems Laboratory (EMSL-LV), Las Vegas, Nevada. The spiking solution will have the following concentrations of unlabelled, internal, and surrogate standards:

2,3,7,8-TCDD - 0.1 ng/ul
$^{13}C_{12}$-2,3,7,8-TCDD - 0.5 ng/ul
$^{37}Cl_4$-2,3,7,8-TCDD - 0.1 ng/ul

When 50 ul of this solution is spiked in 5 grams of soil, the resulting concentrations in the soil are 5 ug/kg of internal standard and 1 ug/kg each of unlabelled and surrogate standard.

NOTE: It is very important that no evaporation of field blank spiking solution be allowed to occur, since the accuracy of results are directly dependent on the addition of a known amount of internal standard.

Solvent

All solvents should be pesticide grade or equivalent. The following solvents will be needed:

Acetonitrile
Dichloromethane
Cyclohexane
Toluene
Benzene
Methanol

Silica Gel

Type 60, 70-230 mesh. Soxhlet extracted with dichloromethane for 24 hours, then activated for 24 hours at 130°C.

Acid Alumina

AG 4, 100-200 mesh. soxhlet extracted with dichloromethane for 24 hours, then activated for 24 hours at 190°C.

Carbopack C

Celite 545

Sodium Sulfate

(ACS) granular, anhydrous.

VII. CALIBRATION AND LIMIT OF DETECTION DETERMINATIONS

Calibration must be done using the internal standard technique. In this case, the internal standard is an isotope of the compound-of-interest, and

metry. The three calibration standard solutions described in section VI are required.

Inject 1-2 ul of each of the calibration standard solutions and acquire selected reaction monitoring data for the following parent- daughter ions:

m/z = 320 →257
m/z = 322 →259
m/z = 328 →263
m/z = 332 →268

For simplicity in subsequent sections, we will refer only to the daughter ions, since quantitation is based on daughter ion response.

Relative response factors for unlabelled 2,3,7,8-TCDD vs the internal standard for triplicate determinations of each of the three calibration standard solutions are calculated.

Equation I: Relative Response Factor (RRFs) for 2,3,7,8-TCDD

$$RRFs = (A_s C_{is})/(A_{is} C_s)$$

where A_s = the sum of the area responses for the ions, m/z 257 and 259, corresponding to the unlabelled standard, 2,3,7,8-TCDD.

A_{is} = the area response of the ion m/z 268, corresponding to the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD.

C_s = the concentration of the unlabelled standard, 2,3,7,8-TCDD

C_{is} = the concentration of the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD.

In the case of the unlabelled 2,3,7,8-TCDD each of the calibration standard solutions must be analyzed in triplicate, and the variation of the RRF values for each compound at each concentration level must not exceed 10% RSD. If the three mean RRF values for each compound do not differ by more than ± 10%, the RRF can be considered to be independent of analyte quantity for the calibration concentration range, and the mean of the three mean RRFs shall be used for concentration calculations. The overall mean is termed a calibration factor.

Similarly, relative response factors for the surrogate standard vs the internal standard for the triplicate determinations of the high level calibration solution are also calculated.

Equation II: Relative Response Factor (RRF_{ss}) for $^{37}Cl_4$-2,3,7,8-TCDD

$$RRF_{ss} = (A_{ss} C_{is})/(A_{is} C_{ss})$$

where A_{ss} = the area response of the daughter ion, m/z 263, corresponding to the surrogate standard, $^{37}Cl_4$-2,3,7,8-TCDD.*

* Subtract 0.01 of any 257 response from the 263 response to correct for contributions of 2,3,7,8-TCDD to the 263 response.

A_{is} = the area response of the ion m/z 268, corresponding to the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD

C_{ss} = the concentration of the surrogate standard, $^{37}Cl_4$-2,3,7,8-TCDD.

and C_{is} = the concentration of the internal standard, $^{13}C_{12}$-2,3,7,8-TCDD.

In the case of the surrogate standard, $^{37}Cl_4$-2,3,7,8-TCDD, the variation of the three RRF values for the high level calibration solution should not exceed 10% RSD. If this is the case, the mean of the three RRFs shall be used for concentration calculations. The overall mean is termed a calibration factor.

The calibration factor for the unlabelled 2,3,7,8-TCDD must be verified on each work shift of 8 hours or less by the analysis of a low level calibration standard. If the RRF for the low level calibration differs from the calibration factor by more than 10%, the entire calibration must be repeated and a new calibration factor determined. The most recently verified calibration factor must be used in all calculations. This verification is only required for the unlabelled standards. There is no need to check the surrogate calibration factor unless the surrogate recoveries appear biased or consistently fall outside the 60-140% control limits.

The theoretical ratio of the m/z 257 to 259 ions for native 2,3,7,8-TCDD is 1.02. However, in practice this ratio will differ from the theoretical due to the very low resolution used in both analyzing quadrupoles for this type of analysis. The ratio must therefore, be determined empirically as follows:

Equation III: (Ratio of native TCDD daughter ions)

$$\text{Ratio} = A_{257}/A_{259}$$

where A_{257} = Area response for ion m/z 257

A_{259} = Area response for ion m/z 259

The mean of the ratios calculated for each of the nine calibration solutions is used for comparison purposes for qualitative identification of 2,3,7,8-TCDD.

It has been found that the sample spiking solution also gives responses for the 257 and 259 daughter ions corresponding to 2,3,7,8-TCDD. These contributions must be subtracted out for each sample. In order to determine this correction factor, add 150 ul of the sample spiking solution to a 5 ml volumetric flask and bring to volume with dichloromethane. Twenty 1-2 ul injections of this solution must be made and the ratio of the area responses for the sum of the m/z 257 and 259 ions vs the m/z 268 ion must be calculated. Twenty separate ratios must be determined.

Equation IV: Blank Response (B) of Sample Spiking Solution

$$B = {}_{b}/A_{is}$$

where A_b = the sum of the area responses for the ions, m/z 257 and 259, obtained with the spiking solution

and A_{is} = The area response of the ion m/z 268, corresponding to the internal standard $^{13}C_{12}$-2,3,7,8-TCDD present in the spiking solution.

The correction factor for the blank contribution to sample response is then calculated as the mean of the 20 blank responses.

Equation V: Correction Factor (C.F.) for Blank Contribution

$$C.F. = \frac{\Sigma B}{n}$$

where ΣB = The sum of the individual blank responses determined by Equation IV.

n = Number of replicate measurements of the blank response (20 are required for initial determination).

Limit Of Detection

The empirical limit of detection will be calculated based on the variability of the blank responses. The blank responses correspond to those obtained from repeat injections of the (diluted) sample spiking solution. Each blank response must be converted to an equivalent concentration of 2,3,7,8-TCDD.

Equation VI: (Conversion of Blank Response to An Equivalent Concentration of 2,3,7,8-TCDD)

$$C_b = \frac{A_b \times Q_{is}}{A_{is} \times RRF_s \times W} = \frac{25 \times A_b}{5 \times A_{is} \times RRF_s}$$

where C_b = equivalent concentration of 2,3,7,8-TCDD in blank (spiking solution) (in units of ug/kg or ppb)

A_b = the sum of the area responses of the ions m/z 257 and 259 for the blank

A_{is} = the area response of the ion m/z 268, corresponding to the internal standard

RRF_s = The relative response factor previously determined for 2,3,7,8-TCDD (Equation I)

Q_{is} = 25 nanograms (the weight of internal standard added to each sample)

W = 5 grams (the weight of wet soil used for each sample)

The standard deviation of the blank responses (in concentration units) must then be calculated.

Equation VII: (Standard Deviation of The Blank Responses)

$$S_b = \frac{(\Sigma C_b^2) - (\Sigma C_b)^2/n}{n-1}$$

where S_b = standard deviation of the blank responses (in units of ug/kg)

C_b = blank response in concentration units (calculated using Equation VI)

n = number of replicate blank results used (20 are required)

Finally, the limit of detection must be calculated from the standard deviation of the blank.

Equation VIII: (Limit of Detection Based on "Well-Known" Blank)*

$$LOD = 2\ t\ S_b$$

where LOD = Limit of Detection

t = the 10% point of the t statistic for a double-sided table with n-1 degrees of freedom (where n is equal to the number of blank results used). NOTE: The LOD must be calculated based on at least 20 replicate blank (i.e. spiking solution) analyses. For n = 20, t = 1.72.

The limit of detection calculated from equation VIII should be less than the required limit of detection of 0.3 ug/kg.

VIII. QUALITY CONTROL REQUIREMENTS

The following quality control (Q.C.) requirements are listed in the order that they must be run. Requirements 1 and 2 are to be run initially before any other samples. Requirements 3 through 7 are the Q.C. samples to be included with each batch of real samples (requirement #8) that is run in one 8-hour time period or on each shift. The requirements 3 through 8 are to be run in the order as they appear in the list below on each shift.

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* Reference - Currie, Lloyd A. "Limits for Qualitative Detection and Quantitative Determination" Anal, Chem., 40, 3, 586-593, 1968

1. An initial calibration must be performed using calibration standard solutions with varied (1, and 25 ug/kg equivalent) native TCDD and 5 ug/kg equivalent internal standard. Calibration for the surrogate standard will be based only on the high level standard (1 ug/kg equivalent). The criteria given in Section VII must be met or the calibration must be repeated.

2. Initially, 20 replicate determinations of the spiking solution must be run and area responses for the sum of m/z 257 and 259 ions vs the m/z 268 ion must be calculated. Twenty separate ratios must be determined (Equation IV) and used in calculating the mean correction factor (Equation V).

3. A 1-point check verification using the 1 ug/kg equivalent native TCDD and 5 ug/kg equivalent internal standard must be run once every 8 hours or on every shift. If the RRF values from this calibration check differ by more than ± 10% from the previously determined mean relative response factor (RRFs), the 3-point calibration must be repeated. The calibration check for the surrogate is not necessary unless the surrogate recoveries appear biased and/or consistently fall outside the 60-140% control limits.

4. A laboratory "method blank" must be run along with each batch of 24 or fewer samples. A method blank is performed by executing all of the specified extraction steps, except for the introduction of a 5 gram sample. The method blank is also dosed with the internal standard and surrogate standard. Results for the method blank must be calculated the same way as samples. This includes correction for the spiking solution contribution as indicated in Equation IX. A positive response $\geq$ 0.3 ug/kg of native TCDD followed by reinjection. If still positive, re-extraction and reanalysis of all related samples must be done.

5. "Field blanks" will be provided to monitor for possible cross-contamination of samples in the lab. The "field blank" will consist of uncontaminated soil (background soil taken off-site). A positive response $\geq$ 0.3 ug/kg native TCDD must be followed by reinjection. If still positive, all samples associated with the field blanks must be re-extracted and reanalyzed.

6. One sample, designated by EPA, must be spiked with native 2,3,7,8-TCDD at a level of 1 ug/kg for each set of 24 or fewer samples. The Field Blank Spiking Solution (Section VI) should be used to spike the designated sample. The recovery must be 0.6 to 1.4 ug/kg or the analysis stopped and all related samples must be re-extracted and reanalyzed.

7. The laboratory will be given performance evaluation samples by EPA to run with each batch of samples. The results from these performance evaluation samples will be evaluated by EPA. If a result is not within the acceptance criteria set by EPA, all samples in the batch associated with that PE sample must be reanalyzed.

8. Each sample must be dosed with 50 ul of the sample spiking solution containing internal standard (equivalent to 5.0 ug/kg) and surrogate standard (equivalent to 1.0 ug/kg). The surrogate recovery must be 0.6 to 1.4 ug/kg or the sample must be reanalyzed.

9. The following qualitative requirements must be met in order to confirm the presence of positive 2,3,7,8-TCDD:

a. The retention time must equal (within 3 seconds) the retention time for the internal standard.

b. The 257/259 ratio must be within the range ± 10% of the value of the ratio determined in Section VII, (Equation III).

c. The ion responses at 257 and 259 must be present and maximize together. The signal to mean noise ratio must be 2.5 to 1 or better for both daughter ions. (Determine the noise level by measuring the random peak to valley signal present on either side [within 20 scans] of the 2,3,7,8-TCDD retention window. The 2,3,7,8-TCDD signal must be at least 2.5 times larger than this.)

d. For those samples giving non-detect results, the result must be less than the 0.3 ug/kg required limit of detection. Otherwise the analysis must be stopped and interferences identified and corrected until the 0.3 ug/kg required limit of detection is met.

e. For each sample, the internal standard must be present with at least a 10 to 1 signal to noise ratio based on the m/z 268 ion response.

IX. SAMPLE COLLECTION, PRESERVATION AND HANDLING

The procedures for sample collection, shipping and handling will be specified by the EPA Regional Office responsible for the monitoring exercise. The sampling team will be provided with an 8 ounce glass jar, and 30-300 grams of soil will be collected. When received in the laboratory, the sample should be thoroughly mixed in the jar for a minimum of 3 minutes, using a stainless steel spatula. The spatula should be used to break up large clumps of soil while mixing to achieve a homogeneous sample.

A 5 gram aliquot sample should be taken and placed in a pre-weighed 10 ml serum vial containing approximately 5 grams of anhydrous sodium sulfate together with a Teflon-faced septum and cap (The entire vial, Na_2SO_4, septum and cap is pre-weighed and labelled). The 5 gram aliquot sample should be representative of the entire sample. Thus, large stones or other particles which are uncharacteristic of the sample, should not be included in the aliquot.

Samples may be stored under ambient conditions as long as temperature extremes (below freezing or above 90°F) are avoided. Samples must be protected from light to avoid photodecomposition.

All samples must be extracted and completely analyzed within 24 hours. Extracts must be held for 6 months prior to disposal.

CAUTION: Although the sample and standards are sealed throughout the extraction procedure, there is always the possibility of leakage and breakage (especially during the sample spiking and centrifuging steps). The analyst should, therefore, be fully protected by wearing plastic gloves and laboratory jacket (a face protector is optional). See Section IV for details on specific safety requirements.

1. Prepare extraction solvent by mixing two volumes acetonitrile with one volume dichloromethane. Mix solvents thoroughly.

2. Weigh the sample vial and determine the net weight of sample (to 3 significant figures).

3. Add 50 ul of the sample spiking solution (containing both internal and surrogate standards). The solution will contain 0.5 ng/ul of internal standard and 0.1 ng/ul of surrogate standard. Add the 50 ul solution directly to the soil, spreading it over several sites on the surface of the soil.

4. Attempt to mix the soil and sodium sulfate by shaking. (Extremely wet samples may not mix well, but DO NOT open the vial to stir the contents.) Additional anhydrous sodium sulfate should be added if needed.

5. Pierce the septum with a disposable needle and leave the needle in place to vent the contents while the extraction solvent is introduced.

6. Add 5 ml of the 2:1 acetonitrile: dichloromethane extraction solvent using a 5 ml syringe and disposable needle. Retain the syringe for solvent additions only.

NOTE: Additional extraction solvent can be added if the analyst judges this necessary to achieve efficient extraction on a particular sample.

7. Remove the syringe and both needles (they should be treated as though contaminated). Dispose of both needles.

8. Shake the vial vigorously on a vortex mixer for 2 minutes.

9. Centrifuge the vial and contents at 4000 rpm for 2 minutes. Remove carefully so as not to disturb the sediment.

10. Insert a needle through the septum so that it just breaks the surface of the septum inside the vial. Using a clean disposable syringe and needle, withdraw approximately 1 ml of the extract; NOTE: The other needle through the septum serves to equilibrate the pressure upon withdrawal of the extract.

11. Invert the syringe and withdraw the plunger to remove the extract from the needle. Dispose of the needle (it is contaminated).

12. Place a 0.45 micron disposable Teflon filter on the syringe and inject the extract into a clean 10 ml serum vial containing 9 ml distilled water. Dispose of the syringe and the filter.

13. Using a Teflon lined septum and an aluminum cap, cover and crimp the vial containing the wat -extract mixture.

14. Manually shake the vial vigorously for about one minute.

15. Centrifuge the vial to separate the dichloromethane phase from the water/acetonitrile phase. The dichloromethane phase will appear as a small bubble at the bottom of the vial.

16. Prepare a miniature drying tube as follows:

a. Plug the tip of a disposable pipet with a small amount of silanized glass wool.

b. Add approximately 1/2 cm anhydrous sodium sulfate.

17. With a disposable syringe and needle, remove the dichloromethane phase from the vial (step 15) as completely as possible.

18. Transfer the dichloromethane phase through the drying tube into a clean 1 ml serum vial.

19. Rinse the drying tube with one-half ml dichloromethane, and collect in the same 1 ml serum vial.

20. Under a stream of nitrogen, evaporate the solvent gently until the volume of solution remaining in the serum vial is 0.05-0.1 ml.

21. Seal the 1 ml serum vial with a Teflon lined septum and cap. Label the vial appropriately.

XI. CLEANUP

The need for cleanup is indicated when a particular extract does not meet the QC criteria for the coelution of all four monitored ions, surrogate recovery, or the ratio A_{257}/A_{259}. Two cleanup procedures are given below.

A. Modified Option A Cleanup

1. Plug the tip of a disposable pipet with a small amount of silanized glass wool.

2. Place approximately a 1 cm layer of silica gel over the glass wool.

3. Place approximately a one-half cm layer of anhydrous sodium sulfate over the silica gel.

4. Plug the tip of a second disposable pipet with a small amount of silanized glass wool.

5. Place approximately 0.5 cm acid alumina over the silanized glass wool.

6. Place approximately 0.5 cm anhydrous sodium sulfate over the alumina.

7. Arrange the two columns so that the silica gel column will elute onto the alumina column, and the alumina column drippings will be collected in a vial.

8. Rinse the two columns with 0.5 ml cyclohexane and discard the eluate.

9. Open the vial containing the extract and add 1 ml cyclohexane to the extract.

10. Under a stream of nitrogen, carefully evaporate the dichloromethane from the extract vial (the volume of the remaining solution should be just under 1 ml).

11. Transfer the entire contents of the extract vial onto the silica column, arranged as specified in step 7.

12. When the solution just reaches the surface of the sodium sulfate layer in the silica gel column, add 0.5 ml cyclohexane.

13. Repeat step 12 a second time. Allow the solution to drip completely after the second addition of cyclohexane.

14. Discard the silica gel column.

15. Rinse the alumina column with an additional 1 ml cyclohexane. Discard the accumulated eluates in the vial beneath the column.

16. Place a clean 1 ml serum vial under the alumina column.

17. Elute the alumina column with three successive portions of 0.5 ml each of 15% by volume dichloromethane in cyclohexane, collecting the eluate in the clean vial.

18. With gentle heating and under a stream of nitrogen, evaporate the solvent until the volume in the vial is 0.05-0.1 ml.

19. Seal the serum vial with a teflon lined septum and cap. Label the vial appropriately. NOTE: If it is a priori known that the second step of cleanup is required, evaporate the sample in stage 18 to just below 1 ml and immediately proceed with a second cleanup as described below.

B. Option D Cleanup

All samples indicating the presence of other TCDD isomers or which contain compounds co-eluting must be cleaned up using Option D.

1. In advance, prepare a mixture of 3.6 g Carbopack C with 16.4 g Celite 545. Activate the mixture at 130°C for 6 hours.

2. Plug the tip of a disposable pipet with a small amount of silanized glass wool.

3. Place 2 cm layer of the carbopack-Celite mixture over the glass wool plug, using suction to pack the column.

4. Rinse the column sequentially with 2 ml toluene, 1 ml dichloromethane-methanol-benzene 75:20:5 by volume), 1 ml cyclohexane-dichloromethane 1:1 by volume), and finally 2 ml cyclohexane. Collect the eluate in a vial and discard the eluate.

5. Dilute the extract which has been cleaned up by the Modified Option A procedure to 1 ml with cyclohexane.

6. Maintaining a discard vial under the column, introduce the extract onto the column.

7. After the solvent has drained, rinse the column successively with 2 ml cyclohexane, 1 ml cyclohexane-dichloromethane mixture (1:1 by volume) and 1 ml dichloromethane-methanol-benzene mixture (75:20:5 by volume).

8. Allow the column to drain completely and discard the accumulated eluates.

9. Place a clean serum vial under the column.

10. Elute the dioxin from the charcoal with 2 ml toluene.

11. With gentle heating and under a stream of nitrogen, concentrate the extract to a volume of 0.05-0.1 ml.

12. Seal the serum vial with a Teflon lined septum and cap. Label appropriately.

XII. GC/MS/MS ANALYSIS

1. Table 1 summarizes the 15 m DB-5 gas chromatographic capillary column and operating conditions. The 15 m DB-5 column has been used for chromatography which is not isomer specific (no valley is observed between the 1,2,3,4-TCDD and 2,3,7,8-TCDD isomers).

2. Standards and samples must be analyzed under identical MS/MS conditions. Selected Reaction Monitoring (SRM) scans are used, using a scan time to give at least five points per chromatographic peak. Recommended MS/MS conditions are given in Table 2.

3. Verify the Calibration of the system daily as described in Section VII. The volume of calibration standard injected should be approximately the same as all sample injection volumes. The requirements described in Section VIII, Parts 9b and 9c must be met for <u>all</u> calibration standards.

4. Inject a 1 to 2 ul aliquot of the sample extract.

5. The presence of TCDD is qualitatively confirmed if the criteria of Section VIII, Part 9, are achieved.

6. For quantitation, measure the area response of the m/z 257 and 259 peaks for 2,3,7,8-TCDD; the m/z 268 peak for $^{13}C_{12}$-2,3,7,8-TCDD, and the m/z 263 peak for $^{37}Cl_4$-2,3,7,8-TCDD. Calculate the concentrations of native and surrogate standards using the following equations:

Equation IX: (Calculation of Concentration of Native 2,3,7,8-TCDD)

$$C_s = \frac{((A_s/A_{is}) - C_F)(Q_{is})}{RRF_s \times W}$$

where C_s = The concentration of native 2,3,7,8-TCDD in ug/kg

A_s = the sum of the area responses for the ions, m/z 257 and 259

A_{is} = the area response for the ion m/z 268

C.F. = correction.factor for spiking solution (blank) previously determined (Equation V)

Q_{is} = quantity (in nanograms) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction

RRF_s = Relative response factor for 2,3,7,8-TCDD calculated previously (Equation I)

W = weight (in grams) of wet soil or sediment sample.

In evaluating the results, a distinction must be made between quantitative measurement and qualitative identification of 2,3,7,8-TCDD. The following steps must be followed in the treatment of all sample results:

1. Calculate the concentration of native 2,3,7,8-TCDD using equation IX.

2. Determine if all of the qualitative identification criteria are met.

3. If all qualitative identification criteria are met, report the concentration found by equation IX, regardless of concentration.

4. If the qualitative identification criteria are not met, and the concentration calculated by equation IX is less than the required limit of detection of 0.3 ug/kg, report the concentration as less than 0.3 ug/kg (i.e. <0.3 ug/kg).

5. If the qualitative identification criteria are not met, and the concentration calculated by equation IX is greater than the required limit of detection of 0.3 ug/kg, the extract must be reinjected. If the qualitative identification criteria are still not met and the result is still greater than 0.3 ug/kg, the extract must be cleaned up or the sample reanalyzed until a satisfactory result is obtained. (i.e. positive result or negative result below 0.3 ug/kg).

NOTE: In reporting results for sample analysis, a comparison is made with the required limit of detection. The limit of detection based on the blank (Equation VIII) might also be used, but interferences may be present and introduce false positives in some cases. However, as explained in Section VII, the empirical limit of detection based on the blank must be less than the required limit of detection of 0.3 ug/kg.

Equation X: (Calculation of concentration of surrogate standard, $^{37}Cl_4$-2,3,7,8-TCDD)

$$C_{ss} = \frac{A_{ss} \times Q_{is}}{A_{is} \times RRF_{ss} \times W}$$

where C_{ss} = the concentration of surrogate standard $^{37}Cl_4$-2,3,7,8-TCDD in ug/kg.

A_{ss} = the area response for the ion m/z 263*

A_{is} = the area response for the ion m/z 268

Q_{is} = quantity (in nanograms) of $^{13}C_{12}$-2,3,7,8-TCDD added to the sample before extraction.

RRF_{ss} = Relative response factor for $^{37}Cl_4$-2,3,7,8-TCDD calculated previously (Equation II).

W = Weight (in grams) of wet soil or sediment sample.

* Subtract 0.0108 of any 257 response from the 263 response to correct for contributions of any 2,3,7,8-TCDD to the 263 response.

Native 2,3,7,8-TCDD contains an innate quantity of $^{37}Cl_4$-2,3,7,8-TCDD. Except at high concentrations of native 2,3,7,8-TCDD, this contribution is too small to significantly affect the calculated concentration of surrogate $^{37}Cl_4$-2,3,7,8-TCDD. The theoretical correction is calculable on the basis of isotope distribution and amounts to 1.08% of the m/z 257 peak. (This correction should be checked at low resolution by analyzing about 200 pg/ul of unlabelled 2,3,7,8-TCDD.) On this basis, the correction to the area count of the surrogate, is made as follows:

$$A_{263} = A_{263} - 0.0108\ A_{257}$$

Calculate the analytical percent recovery of the surrogate standard.

$$\text{Surrogate Analytical Percent Recovery} = \frac{\text{amount measured* (nanograms)}}{5 \text{ ng}} \times 100$$

* NOTE: The amount measured is equal to the concentration found by equation X multiplied by the weight of soil used for the sample (i.e., $C_{ss} \times W$).

XIII. METHOD PERFORMANCE

The required detection limit for this method is 0.3 ug/kg. For certain samples, this detection limit may not be achievable because of interferences. These samples require cleanup as described in Section XI. This method has been compared with the EPA-IFB GC/MS Method for 2,3,7,8-TCDD and found to be applicable to analyses of soils where 2,3,7,8-TCDD is the only tetrachloro isomer known to be present.

TABLE I

OPERATING CONDITIONS FOR DB-5 GAS CHROMATOGRAPHY COLUMN

COLUMN	DB-5
Length	15 m
I. D.	0.32 mm
Film Thickness	1.0 micron
2,3,7,8-TCDD R. T. (approx.)	5-6 min.
Carrier gas	N_2
Initial Temperature	150°C
Initial Time	1.0 min.
Splitless Time	1.0 min.
Program Rate	20°C/min.
Final Temperature	240°C
Split Flow	20 ml/min.
Septum Purge Flow	0.6 ml/min.
Capillary Head Pressure	8 psi
Transfer Line Temperature	240°C

Instrument	TAGA® or TAGA® 6000E
Ion Source	Townsend/glow discharge CI
CI Reagent Gas	Zero grade air (H_2 and He free)
Reagent Gas Flow	35 ± ml/min.
Source Temperature	200°C
Discharge Current	-1 mA
Q1 Resolution	3 amu at 50% peak height at m/z = 320 (single MS)
Q3 Resolution	3 amu at 50% peak height at m/z = 320 (single MS)
Collision Energy (LAB)	55eV [(OR + GR)/2-R2] or 55eV (OR - R_2)
Collision Gas	Ar
Collision Gas Thickness	400 x 10^{12} molecules/cm^2

Ions Monitored:	Q_2	Q_3
	320	257 (native-TCDD)
	322	259 (native-TCDD)
	328	263 (surrogate standard)
	332	268 (internal standard)

XIV. DATA REPORTING

Report all data in units of micrograms per kilogram of wet soil. Use three significant figures at concentrations above 1 ug/kg and 2 significant figures at concentrations below 1 ug/kg. The data package must include the following information:

1. Individual and mean response factor for the three-point calibration of unlabelled 2,3,7,8-TCDD. (Based on High level standard only).

2. Individual and mean response factors for the isotopic surrogate standard (based on high level standard only).

3. The individual ratios of the sum of areas 257 and 259 ions to the 268 ion for 20 replicate measurements of the blank (i.e., sample spiking solution), and the mean Correction Factor based on these ratios.

4. The empirical limit of detection based on the 20 blank measurements.

5. The daily or shift verification of the mean response factors.

6. The percent accuracy i.e., (analytical percent recovery) for the surrogate standard.

7. The result for the method blank.

8. The percent recovery of native TCDD from the spiked sample.

9. The result for the PE sample

10. The result for the field blank.

11. The data filename (to facilitate data retrieval).

12. The sample identification number (as assigned by the field sampling team).

13. Analytical date and time.

14. The area responses for ions 257, 259, 263, and 268.

15. The observed response ratio of ions 257/259 for the sample.

16. The calculated value for native 2,3,7,8-TCDD. (Values above or below 0.3 ug/kg are to be reported only if qualitative identification criteria are met.)

17. If no 2,3,7,8-TCDD was detected, report "not detected" or N.D. and the 0.3 ug/kg required detection limit.

18. The mass chromatograms for all samples and standards. Include both the real-time display data and reduced data showing limits of integration. Include any computer generated response tables.

19. The weight of the original wet sample aliquot.

20. Documentation on the source and history of the native and labelled 2,3,7,8-TCDD standards used.

21. Any other supporting documentation. An example of the required data format follows:

ION	ION 257	ION 259	ION 268	BLANK RESPONSE	MA.. CONC
1					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

L.F.
L03

0 1180 08

IVE RF	NATIVE CONC	1	LAB:
ROGATE RF *	SU[illegible]ATE CONC	0.3	DATE:
N RATIO	I STD CONC	5	CASE NO:
TION FACTOR			

SIS	ANALYSIS TIME	ION 257	ION 259	ION 263	ION 268	RATIO 257/259	RF NATIVE	NAT RF % DIFF	RF SURROGATE	RF SURR %DIFF	COMMENTS (25 CHAR MAX)
E											

0 1 3 0 4

NATIVE RESPONSE FACTOR OUTSIDE LIMITS *
NATIVE ION RATIO OUTSIDE LIMITS

SURROGATE CONC
I STD CONC
RF NATIVE
RF SURROGATE
ION RATIO AVERAGE
CORRECTION FACTOR

E	SMO	EPA	ANALYSIS	WET WT	ION	ION	ION	ION	RATIO	SURR	UG/KG
LE #	SAMPLE #	SAMPLE #	DATE - TIME	GRAMS	257	259	263	268	257/259	ACC	TCDD

SMO PLE NO	EPA SAMPLE NO	ANALYSIS DATE	ANALYSIS TIME	MATIVE RATIO	SURROGATE % ACC	TCDD CONC	DL	COMMENTS

SURROGATE PERCENT ACCURACY OUTSIDE LIMITS
DL HIGH DETECTION LIMIT

Calculations:

* Note: The equivalent concentrations of 2,3,7,8-TCDD (last column) are calculated using Equation VI

$$C_b = \frac{A_b \circ Q_{is}}{A_{is} \circ RRF_s \circ W}$$

Other calculations required are:

1. Equation V: Correction Factor (C.F.) for Blank Contribution

$$C.F. = \frac{\Sigma B}{n} = \underline{\qquad\qquad}$$

2. Equation VII: (Standard Deviation of the Blank Responses)

$$S_b = \frac{\left(\Sigma C_b^2 \right) - \left(\Sigma C_b \right)^2 / n}{n-1} \qquad \underline{\qquad\qquad}$$

3. Equation VIII: (Limit of Detection based on "Well Known" Blank)

$$LOD = 2 \circ t \circ S_b = \underline{\qquad\qquad}$$

1.2 QUALITY CONTROL

The procedures indicated below are to be performed for all analyses. Specific instructions relevant to particular analyses are given in the pertinent analytical procedures.

1.2.1 Field Quality Control

The sampling component of the Quality Assurance Project Plan (QAPP) shall include:

Reference to or incorporation of accepted sampling techniques in the sampling plan;

Procedures for documenting and justifying any field actions contrary to the QAPP;

Documentation of all pre-field activities such as equipment check-out, calibrations, and container storage and preparation;

Documentation of field measurement quality control data (quality control procedures for such measurements shall be equivalent to corresponding laboratory QC procedures);

Documentation of field activities;

Documentation of post-field activities including sample shipment and receipt, field team de-briefing and equipment check-in;

Generation of quality control samples including duplicate samples, field blanks, equipment blanks, and trip blanks; and

The use of these samples in the context of data evaluation, with details of the methods employed (including statistical methods) and of the criteria upon which the information generated will be judged.

1.2.2 Analytical Quality Control

A quality control operation or component is only useful if it can be measured or documented. The following components of analytical quality control are related to the analytical batch. The procedures described are intended to be applied to chemical analytical procedures; although the principles are applicable to radio-chemical or biological analysis, the procedures may not be directly applicable to such techniques.

All quality control data and records required by this section shall be retained by the laboratory and shall be made available to the data requestor as appropriate. The frequencies of these procedures shall be as stated below or at least once with each analytical batch.

1.2.2.1 Spikes, Blanks and Duplicates

General Requirements

These procedures shall be performed at least once with each analytical batch with a minimum of once per twenty samples.

1.2.2.1.1 Duplicate Spike

A split/spiked field sample shall be analyzed with every analytical batch or once in twenty samples, whichever is the greater frequency. Analytes stipulated by the analytical method, by applicable regulations, or by other specific requirements must be spiked into the sample. Selection of the sample to be spiked and/or split depends on the information required and the variety of conditions within a typical matrix. In some situations, requirements of the site being sampled may dictate that the sampling team select a sample to be spiked and split based on a pre-visit evaluation or the on-site inspection. This does not preclude the laboratory's spiking a sample of its own selection as well. In other situations the laboratory may select the appropriate sample. The laboratory's selection should be guided by the objective of spiking, which is to determine the extent of matrix bias or interference on analyte recovery and sample-to-sample precision. For soil/sediment samples, spiking is performed at approximately 3 ppm and, therefore, compounds in excess of this concentration in the sample may cause interferences for the determination of the spiked analytes.

1.2.2.1.2 Blanks

Each batch shall be accompanied by a reagent blank. The reagent blank shall be carried through the entire analytical procedure.

1.2.2.1.3 Field Samples/Surrogate Compounds

Every blank, standard, and environmental sample (including matrix spike/matrix duplicate samples) shall be spiked with surrogate compounds prior to purging or extraction. Surrogates shall be spiked into samples according to the appropriate analytical methods. Surrogate spike recoveries shall fall within the control limits set by the laboratory (in accordance with procedures specified in the method or within ±20%) for samples falling within the quantification limits without dilution. Dilution of samples to bring the analyte concentration into the linear range of calibration may dilute the surrogates below the quantification limit; evaluation of analytical quality then will rely on the quality control embodied in the check, spiked and duplicate spiked samples.

1.2.2.1.4 Check Sample

Each analytical batch shall contain a check sample. The analytes employed shall be a representative subset of the analytes to be determined. The concentrations of these analytes shall approach the estimated quantification limit in the matrix of the check sample. In particular, check samples for metallic analytes shall be matched to field samples in phase and in general matrix composition.

1.2.2.2 Clean-Ups

Quality control procedures described here are intended for adsorbent chromatography and back extractions applied to organic extracts. All batches of adsorbents (Florisil, alumina, silica gel, etc.) prepared for use shall be checked for analyte recovery by running the elution pattern with standards as a column check. The elution pattern shall be optimized for maximum recovery of analytes and maximum rejection of contaminants.

1.2.2.2.1 Column Check Sample

The elution pattern shall be reconfirmed with a column check of standard compounds after activating or deactivating a batch of adsorbent. These compounds shall be representative of each elution fraction. Recovery as specified in the methods is considered an acceptable column check. A result lower than specified indicates that the procedure is not acceptable or has been misapplied.

1.2.2.2.2 Column Check Sample Blank

The check blank shall be run after activating or deactivating a batch of adsorbent.

1.2.2.3 Determinations

1.2.2.3.1 Instrument Adjustment: Tuning, Alignment, etc.

Requirements and procedures are instrument- and method-specific. Analytical instrumentation shall be tuned and aligned in accordance with requirements which are specific to the instrumentation procedures employed. Individual determinative procedures shall be consulted. Criteria for initial conditions and for continuing confirmation conditions for methods within this manual are found in the appropriate procedures.

1.2.2.3.2 Calibration

Analytical instrumentation shall be calibrated in accordance with requirements which are specific to the instrumentation and procedures employed. Introductory Methods 7000 and 8000 and appropriate analytical procedures shall be consulted for criteria for initial and continuing calibration.

1.2.2.3.3 Additional QC Requirements for Inorganic Analysis

Standard curves used in the determination of inorganic analytes shall be prepared as follows:

Standard curves derived from data consisting of one reagent blank and four concentrations shall be prepared for each analyte. The response for each prepared standard shall be based upon the average of three replicate readings of each standard. The standard curve shall be used with each subsequent analysis provided that the standard curve is verified by using at least one reagent blank and one standard at a level normally encountered or expected in such samples. The response for each standard shall be based upon the average of three replicate readings of the standard. If the results of the verification are not within ±10% of the original curve, a new standard shall be prepared and analyzed. If the results of the second verification are not within ±10% of the original standard curve, a reference standard should be employed to determine if the discrepancy is with the standard or with the instrument. New standards should also be prepared on a quarterly basis at a minimum. All data used in drawing or describing the curve shall be so indicated on the curve or its description. A record shall be made of the verification.

Standard deviations and relative standard deviations shall be calculated for the percent recovery of analytes from the spiked sample duplicates and from the check samples. These values shall be established for the twenty most recent determinations in each category.

1.2.2.3.4 Additional Quality Control Requirements for Organic Analysis

The following requirements shall be applied to the analysis of samples by gas chromatography, liquid chromatography and gas chromatography/mass spectrometry.

The calibration of each instrument shall be verified at frequencies specified in the methods. A new standard curve must be prepared as specified in the methods.

The tune of each GC/MS system used for the determination of organic analytes shall be checked with 4-bromofluorobenzene (BFB) for determinations of volatiles and with decafluorotriphenylphosphine (DFTPP) for determinations of semi-volatiles. The required ion abundance criteria shall be met before determination of any analytes. If the system does not meet the required specification for one or more of the required ions, the instrument must be retuned and rechecked before proceeding with sample analysis. The tune performance check criteria must be achieved daily or for each 12 hour operating period, whichever is more frequent.

Background subtraction should be straightforward and designed only to eliminate column bleed or instrument background ions. Background subtraction

actions resulting in spectral distortions for the sole purpose of meeting special requirements are contrary to the objectives of Quality Assurance and are unacceptable.

For determinations by HPLC or GC, the instrument calibration shall be verified as specified in the methods.

1.2.2.3.5 Identification

Identification of all analytes must be accomplished with an authentic standard of the analyte. When authentic standards are not available, identification is tentative.

For gas chromatographic determinations of specific analytes, the relative retention time of the unknown must be compared with that of an authentic standard. For compound confirmation, a sample and standard shall be re-analyzed on a column of different selectivity to obtain a second characteristic relative retention time. Peaks must elute within daily retention time windows to be declared a tentative or confirmed identification.

For gas chromatographic/mass spectrometric determinations of specific analytes, the spectrum of the analyte should conform to a literature representation of the spectrum or to a spectrum of the authentic standard obtained after satisfactory tuning of the mass spectrometer and within the same twelve-hour working shift as the analytical spectrum. The appropriate analytical methods should be consulted for specific criteria for matching the mass spectra, relative response factors, and relative retention times to those of authentic standards.

1.2.2.3.6 Quantification

The procedures for quantification of analytes are discussed in the appropriate general procedures (7000, 8000) and the specific analytical methods.

In some situations in the course of determining metal analytes, matrix-matched calibration standards may be required. These standards shall be composed of the pure reagent, approximation of the matrix, and reagent addition of major interferents in the samples. This will be stipulated in the procedures.

Estimation of the concentration of an organic compound not contained within the calibration standard may be accomplished by comparing mass spectral response of the compound with that of an internal standard. The procedure is specified in the methods.

1.3 DETECTION LIMIT AND QUANTIFICATION LIMIT

The detection limit and quantification limit of analytes shall be evaluated by determining the noise level of response for each sample in the batch. If analyte is present, the noise level adjacent in retention time to the analyte peak may be used. For wave-length dispersive instrumentation, multiple determinations of digestates with no detectable analyte may be used to establish the noise level. The method of standard additions should then be used to determine the calibration curve using one digestate or extracted sample in which the analyte was not detected. The slope of the calibration curve, m, should be calculated using the following relations:

m = slope of calibration line

S_B = standard deviation of the average noise level

$MDL = KS_B/m$

For K = 3; MDL = method detection limit.

For K = 5; MQL = method quantitation limit.

1.4 DATA REPORTING

The requirement of reporting analytical results on a wet-weight or a dry-weight basis is dictated by factors such as: sample matrix; program or regulatory requirement; and objectives of the analysis.

Analytical results shall be reported with the percent moisture or percent solid content of the sample.

1.5 QUALITY CONTROL DOCUMENTATION

The following sections list the QC documentation which comprises the complete analytical package. This package should be obtained from the data generator upon request. These forms, or adaptations of these forms, shall be used by the data generator/reportor for inorganics (I), or for organics (O) or both (I/O) types of determinations.

1.5.1 Analytical Results (I/O: Form I)

Analyte concentration.

Sample weight.

Percent water (for non-aqueous samples when specified).

Final volume of extract or diluted sample.

Holding times (I: Form X).

1.5.2 Calibration (I: Form II; O: Form V, VI, VII, IX)

Calibration curve or coefficients of the linear equation which describes the calibration curve.

Correlation coefficient of the linear calibration.

Concentration/response data (or relative response data) of the calibration check standards, along with dates on which they were analytically determined.

1.5.3 Column Check (O: Form X)

Results of column chromatography check, with the chromatogram.

1.5.4 Extraction/Digestion (I/O: Form I)

Date of the extraction for each sample.

1.5.5 Surrogates (O: Form II)

Amount of surrogate spiked, and percent recovery of each surrogate.

1.5.6 Matrix/Duplicate Spikes (I: Form V, VI; O: Form III)

Amount spiked, percent recovery, and relative percent difference for each compound in the spiked samples for the analytical batch.

1.5.7 Check Sample (I: Form VII; O: Form VIII)

Amount spiked, and percent recovery of each compound spiked.

1.5.8 Blank (I: Form III; O: Form IV)

Identity and amount of each constituent.

1.5.9 Chromatograms (for organic analysis)

All chromatograms for reported results, properly labeled with:

- Sample identification
- Method identification
- Identification of retention time of analyte on the chromatograms.

1.5.10 Quantitative Chromatogram Report (O: Forms VIII, IX, X)

Retention time of analyte.

Amount injected.

Area of appropriate calculation of detection response.

Amount of analyte found.

Date and time of injection.

1.5.11 Mass Spectrum

Spectra of standards generated from authentic standards (one for each report for each compound detected).

Spectra of analytes from actual analyses.

Spectrometer identifier.

1.5.12 Metal Interference Check Sample Results (I: Form IV)

1.5.13 Detection Limit (I: Form VII; O: Form I)

Analyte detection limits with methods of estimation.

1.5.14 Results of Standard Additions (I: Form VIII)

1.5.15 Results of Serial Dilutions (I: Form IX)

1.5.16 Instrument Detection Limits (I: Form XI)

1.5.17 ICP Interelement Correction Factors and ICP Linear Ranges (when applicable) (I: Form XII, Form XIII).

1.6 REFERENCES

1. Guidelines and Specifications for Preparing Quality Assurance Program Plans, September 20, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, QAMS-004/80, Washington, DC 20460.

2. Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans, December 29, 1980, Office of Monitoring Systems and Quality Assurance, ORD, U.S. EPA, QAMS-005/80, Washington, DC 20460.

Draft

Regional Technical Assistance for Preparing Quality Assurance Project and Laboratory Plans

ROQA-005/85
(Revised Jan, 1986)

by

Steven R. Lemons

Office of Quality Assurance
Environmental Services Division
U.S. Environmental Protection Agency, Region VI

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I. Who must prepare a Quality Assurance Project Plan (QAPjP)

The U.S. EPA Quality Assurance (QA) program embraces many functions including: establishing QA policy and guidelines for development of program and project operational plans; establishing criteria and guidelines for assessing data quality; serving as a QA information focal point; auditing to ascertain effectiveness of QA implementation; and identifying and developing QA training programs.

The goals and policy of EPA's QA program is to ensure that all environmentally related measurements (data collection activities) regulated and supported by or for EPA produce data of known quality. The quality of data is known when all components associated with its derivation are thoroughly documented, such documentation being verifiable and defensible. Verifiable is defined as the ability to prove or substantiate any claim or result related to the documented record. Defensible is defined as the ability to withstand any reasonable challenge related to veracity or truthfulness.

In order to establish quality assurance solidly in all data collection activities U.S. EPA issued Order 5360.1. This order establishes policy and program requirements for the conduct of quality assurance (QA) for all environmentally related measurements performed by or for the Agency.

To ensure that all environmentally related measurements (data collection activities) meets U.S. EPA Quality Assurance Policy and requirements, the following organizations should develop and implement a Quality Assurance Project and/or laboratory plan:

* EPA Regional Program Offices (primarily special projects)
* EPA's contractors
* State Agencies
  State contractors
* NPDES & POTW Permittees
* RCRA Permittees
* Laboratories preforming analytical services (directly or indirectly) for support of programs regulated by U.S. EPA
* Other organizations under formalized agreements.

Some laboratories may integrate their QA Plan into specific QA Project Plans (ie. State Laboratories, Permittee's laboratories, and Superfund contractors). However, our office recommends that all laboratories prepare and maintain a laboratory QA plan.

II. Where and how can QA Project Plan be integrated.

Listed below are several options that can be employed by preparers.

Option A: A separate identifiable QA Project Plan.

* Option B: The QA Project Plan can be integrated with Work Plans.

* Option C: The QA Project Plan can be integrated with Waste Analysis Plans (RCRA Permittees).

* Option D: The QA Project Plan can be integrated with Permits POTW, NPDES and RCRA Permittees).

What ever option is choosen the QA Project Plan must meet the minimal requirements as set forth in this guidance document.

* Whenever this option is chosen a "QA Projected Plan locator page" must be inserted in the table of contents of the document.

IIa. For laboratories:

A separate identifiable Laboratory QA Plan should be prepared and maintained at the facility.

III. Document Control

Purpose: Document control will serve to provide

- an easy, convenient way of preparing the QA Project, laboratory plans, and standard operating procedures.
- A easy way to revise and update the elements of QA plans and standard operating procedures.
- a focal point for internal/external audits and inspections.
- reviewers and inspectors a means by which deficiencies and corrective actions can easily be referenced in reports.

Minimum Requirements:

All Quality Assurance Project, Laboratory plans and standard operating procedures must be prepared using a document control format consisting of following information placed in the upper right-hand corner of each document page:

- Section Number _____
- Revision Number _____
- Date (of revision) _____
- Page _____ of _____

IV. Standard Operating Procedures (SOPs)

Purpose: Generally, simply citing a published method is not adequate. Published methods rarely have all of procedural details, and those that do generally have to be modified for the application or facilities at hand. The development of SOPs are fundamental for review and laboratory audit/inspection processses.

Developing SOPs:

Standard Operating Procedures (SOPs) shall be detailed documents describing who does what, when, where, how and why, in a stepwise manner. These SOPs shall be consistent with National SOPs endorsed or issued by Program or Regional Offices. They shall be sufficiently complete and detailed to ensure:

1. Data of known quality and intergrity are collected to meet the monitoring objectives.

2. The minimum loss of data due to out-of-control conditions.

SOPs shall be:

1. Adequate to establish traceability of standards, instrumentation, samples, and environmental data.

2. Simple, so a user with basic education, experience and/or training can properly use them.

3. Complete enough so the user/reader follows the directions in a stepwise manner through the sampling, analysis, and data handling process.

4. Consistent with sound scientific/engineering principles.

5. Consistent with current EPA regulations and guidelines.

6. Consistent with the manufacturer's specific instrumentation manuals.

SOPs shall provide for documentation sufficiently complete to:

1. Record the performance of all tasks and their results.

2. Explain the cause for missing data.

3. Demonstrate the validation of data each time they are recorded, calculated, or transcribed.

SOPS should be addressed in all QA Project or Laboratory Plan as outlined below:

° Standard Operating Procedures (SOPs) must be prepared for all routinely used sampling, analytical and management methods or protocols.

° SOPs must meet the minimum criteria as identified in "Developing SOPs" (See previous section).

° In cases where published methods have all the procedural details, with little or no modifications, photocoping the appropriate procedures will normally be adequate. However, it must meet the minimum criteria as identified in "Developing SOPs".

° In either case (development of specific SOPs or photocoping of published methods), the SOPs must be

- documented (using document control format)
- reviewed annually
- contain a cover page indicating who reviewed the SOP and the data of review.

° To accomplish these objectives, SOPs should address the following types of items:

1. General network design.
2. Specific sampling-site selection.
3. Sampling and analytical methodology.
4. Probes, collection devices, storage containers, and sample additives such as preservatives.
5. Special precautions, such as holding times and protection from heat, light, reactivity, and combustibility.
6. Federal reference, equivalent, and alternate test procedures.
7. Instrumentation selection and use.
8. Calibration and standardization.
9. Preventive and remedial maintenance.
10. Replicate sampling and analysis.

11. Blind and spiked samples.
12. Quality control procedures such as inter- and intra- field or laboratory activities.
13. Documentation procedures.
14. Sample custody and handling procedures.
15. Sample transportation procedures.
16. Safety.
17. Data handing/evaluation procedures.
18. Precision, accuracy, completeness, representativeness, and comparability procedures (control charts, calculations, statistical tests, etc.).
19. Service contracts.
20. Document control.
21. Corrective action procedures.

Element 1. Title Page (For both QA Project and Laboratory Plans)

Purpose: Primarily the title page provides a means of identifing the organization responsible for preparing the QA Plan and it will serve as documentary evidence that all appropriate responsible individuals have reviewed and approved the QA Plan. It will also serve to document the date of approval and provide a means of tracking the review and approval process.

Minimum Requirements:

The following information must be included on the title page:

° The title/name of the Project, Facility, or Laboratory.

° The name of the organization that is responsible for the Quality Assurance of the Project, Facility, or Laboratory.

° *If a contractor is preparing the QA Project Plan for an organization* (see above), then the contractor also must be identified.

° At the bottom of the title page, provisions must be made for the signatures of approving personnel.

QA Project Plans:

a) For in-house projects
   1) Project Officer
   2) QA Officer (not from from the Office of Quality Assurance)
   3) Robert G. Forrest, Chief Office of Quality Assurance U.S. EPA Region VI.
   optional 4) Laboratory personnel (Directors, Section Chiefs, QA Lab Officials).

b) For State and EPA Contractors (i.e. CERCLA)
   1) The organization's Project Manager/Officer
   2) The organization's QA Official
   3) EPA's Project Officer
   4) Robert G. Forrest, Chief Office of Quality Assurance U.S. EPA Region VI
   optional 5) Laboratory personnel (Directors, Section Chiefs, QA Lab Officials).

c) For State Contractors

1) Contractor's Project Manager/Officer
2) Contractor's QA Official
3) State Agency's Project Manager/Officer
4) State Agency's Project QA Official
5) EPA's Project Officer
6) Robert G. Forrest, Chief
Office of Quality Assurance
U.S. EPA Region VI
Optional 7) Laboratory personnel (Lab Directors, Section Chiefs, etc.)

d) For Permittees

1) Permittee's Project Manager/Officer
2) Permittee's QA Offical
Optional 3) State/City Project Manager/Officer
Optional 4) State/City QA Offical
Optional 5) EPA Project Officer
Optional 6) Robert G. Forrest, Chief
Office of Quality Assurance
U.S. EPA Region VI

Laboratory QA Plans:

a) For State Laboratories

1) Laboratory Director/Manager
2) Laboratory QA Offical
3) State Agency QA Offical
Optional 4) Laboratory Section Chiefs

b) For Commercial and Permittee Laboratories

1) Laboratory Directors/Managers
2) Laboratory QA Offical
3) Laboratory Section Chiefs
4) Permittee's Program Managers

Element 2. Table of Contents

The QA Project Plan Table of contents must address each of the following:

° A serial listing of each of the 16 QA project plan elements* (components).

° A listing of any appendices which are required to augment (to facilitate complete review) the QA Project Plan as presented (i.e., standard operating procedures, field manuals, work plans, operations plans, etc.)

° At the end of the Table of Contents, list the individuals on the title page and any other individuals (i.e., contracted lab) receiving official copies of the QA Project and any subsequent revisions.

- The individuals responsible for distributing the QA Project Plan and any subsequent revisions.

a) For EPA in-house projects
1.) EPA Project Officer

b) For State Agencies
1) The State Project Officer

c) For Contractors/Permittees
1) The Organization's Project Managers

d) For Laboratories
1) The Laboratory's Directors/Managers

° Serial listing of all 16 quality assurance project plan component is required, as listed below. Each component must be included and addressed for each project plan.

1) Title page with provision for approval signatures

2) Table of contents

3) Project description

4) Project organization and responsibility

5) Data quality objectives for measurement data in terms of precision, accuracy, completeness, representativeness and comparability

5a) Laboratory Data Quality Objectives*

6) Sampling procedures

6a) Good Laboratory Practices*

7) Sampling custody

8) Calibration procedures and frequency

9) Analytical procedures

10) Data reduction, validation and reporting

11) Internal quality control checks and frequency

12) Management, performance, technical systems, and data quality audits, and frequency

13) Preventive maintnance procedures and schedules

14) Specific routine procedures to be used to assess data precision, accuracy and completeness of specific measurement parameters involved

15) Corrective action

16) Quality assurance reports to management

*For Laboratory's not intergrated in a formal QA Project Plan.

- The serial listing of each of the 16 QA project plan elements (components) are the same, except number 5 which should be entitled "Laboratory Data Quality Objectives" and number 6 which should be entitled "Good Laboratory Practices (GLP)".

- If a laboratory also performs field activities then number 6 Sample Procedures must be addressed and number 6a will be added, to address Good Laboratory Practices (GLP).

Element 3 Project Description

Purpose: To provide sufficient information (on a project) for integration into and evaluation of the remaning elements (components) of the QA Project Plan. It must be complete enough to evaluate the appropriateness of Data Quality Objectives, sampling design, sampling and analytical methods, etc.

For Projects

This Element should address the following items:

A. Background Information and Previous Data Assessments

- A comprehensive (chronological) discussion of the project/site history, environmental setting (physiography, geology, hydrogeology, etc.), summary resultrs of data previously collected (chemical, biological, and physical parameters; matrices, etc.) previous data assessments (statistical results), summary of previous QA reports, and any other QA related information (i.e. previous data quality objectives, previous project goals).

B. Project Objectives (Purpose) and Scope

- A comprehensive statement addressing the project's objective (purpose). This item can be addressed in Element 5, if so, please reference Element 5 in this section.

D. Revisions (continuous projects)

- This element should be revised annually to provide updated information and changes. This element will require the inclusion of Parts A & B (above) of the previous year.

C. Schedule of Tasks and Milestones

- Both activities and milestones need to be stated in specific and measureable terms, so their timely attainment or non-attainment can be easily observed and documented.

- This item should consist of a list of activities and milestones which will lead to the accomplishment of the project purpose (objectives).

For example:

- dates anticipated for start and completion of the project,
- initiation of sample collection,
- sample analysis, data review and reporting,
- data validation and data assessments,
- final QA report preparation, and
- other applicable activities.

E. Data Usage. (can be addressed in Element 5)

This section should consist of a statement outlining the intended data usage so that appropriate review and evaluation can be made on the Data Quality Objectives, sampling and analytical methods, and any other QA/QC components of the QA Project Plan, When applicable, secondary uses of the data should also be identified.

For Laboratories

The following items should be addressed:

A. A comprehensive discussion of the laboratory's overall objective/purpose of thiS QA Program. Our office recommends that specific Laboratory/company policies be devloped and documents.

Some examples are listed below:

° To maintain an effective, routine quality control program to measure and verify laboratory performance.

° To meet data quality requirements for accuracy, precision and completeness through the use of proven or recommended methodologies.

° To provide sufficient flexibility to meet specific data quality requirements.

° To identify and provide corrective actions as soon as possible to avoid any possible adverse affect on data quality.

° To monitor and assess the operational performance of the laboratory on a routine basis including internal and external audits.

° Maintain complete written records of documentation chain-of-custody, analytical SOPs, calibration and preventive maintance SOPs, data validation and reduction procedures, etc.

° Other items that laboratories should address include:

- resources to maintain QA
- document control
- external review of QA program
- etc.

PROJECT ORGANIZATION AND RESPONSIBILITY

Purpose: To provide documentory evidence of what inter- and intraorganizations are participating in the QA Project or Laboratory plan. It serves to identify the individuals within each organization who are responsible for Quality Assurance (both program personnel and QA Office/ Officer). It also provides as a means for tracking, auditing, assessing training needs, and for developing and improving QA planning.

There are two distinct lines of responsibilities: a) the program/laboratory/facility personnel and b) the QA officer. The decision makers and resource manager's responsibilities within programs offices, laboratories/facilities must be documented. Because of management responsibilities to making decisions and allocation of resources, they must be responsible for the quality of data, equipment/instruments, facilities and field/laboratory functions. QA Officer's responsibilities must also be identify and document in order to reduce biases and provide the necessary external quality control assessments of QA Plans.

Minimum Requirements:

- This element must clearly identify and document all inter- and intra-organizations (i.e. contractors, labs) that are participating in each project.
- For each organization that is identified, individuals must be identified (including all laboratory sample custodians) by name and his/her responsibilities must be documented.
  - Must include the Project or Laboratory QA Officer's responsibilities.
  - Must include program/management personnel responsiblities.
- The QA project plan should contain a flow chart identfying the organizations and line of authority.

Below are some previous program personnel and QA officers responsibilities that should be developed and documented in each QA Plan (Project and Laboratory). These include, but not limited to:

Program/Project Manager Responsibilities

- Ensure Subcontractor Procurement meet QA/QC requirements
- Assignment of duties of the Project (lab.) Staff and orientation of the staff to the QA needs and requirements of the project (lab).
- Ensure all approved project-specific (lab-specific) procedures and internally prepared plans, drawings and reports meet QA drequirements.
- Serve as liasion (with QA official) between the Project Staff and other internal or external orgnanizations or organizational sub-units.
- Serve as the "collection point." for Project Staff reporting of noncomformances and changes in QA project documents and activities.
- Other

Field Coordinator Responsibilities (Lab. Dept. or Section Heads)

- Will be responsible for all field activities including those of subcontractors.
- Ensure that all field equipment/instrument meet performance criteria and calibration requirements
- Ensure proper labeling, handling, storage, and shipping requirements have been meet.
- Ensure all appropriate chain-of-custody procedures have been followed.
- Assist the QA Official in implementing any field audits.
- Will coordinate with line management and QA Official the procurement and control of equipment/instruments to ensure they meet QA or QC requirements of the project (or Laboratory).
- Other

Laboratory Director/Manager Responsibilities

- ° General supervision of laboratories
- ° Collaboration with the Project Manager (Permittee) in establishing quality sampling and testing programs.
- ° Schedule and execution of testing program.
- ° Serve as liasion between the Laboratory Staffs and other groups
- ° Serves as the "collectin point" for Laboratory Staff reporting of nonconformances and changes in laboratory activities
- ° Notification of the Laboratory and Quality Assurance Groups of specific laboratory nonconformances and changes
- ° Maintenance of laboratory data and checkprints while the project, or testing phase, is in progress
- ° Relese of testing data and results
- ° Calibration of equipment
- ° Storage of samples.

QA Officer Responsibilities:

a. Be the official organizational contact for all QA matters for the project. For example QA project plan implementation, sampling and analytical methodologies, Data Quality Objectives (DQOs), field and laboratory audits, management and data quality audits, PE and QC studies, etc.

b. Actively identify and respond to QA needs, resolve problems, and answer requests for guidance or assistance. For example field sampling problems (limited supplies of sample container's), transportation problems (holding time conflicts), etc.

c. Review, evaluate and approve QA project plans prior to our office (EPA Region 6 Office of Quality Assurance) review, evaluation and approval/non-approval.

d. Provide guidance in the development of QA project plans to each respective organizations program offices, management offices and project/program managers or officers.

e. Ensure that management, data quality, field and laboratory audits are performed on QA Project Plans.

f. Actively track the progress of all QA tasks in Project Plans (from preplanning to data assessments) and consult periodically with program/project managers.

g. Prepare and submit all QA reports (with recommendations and comments) to the appropriate line managers in their organization and to EPA officials.

h. Assure that appropriate corrective actions are taken on all QA tasks when, where and however needed.

i. Ensure that data of known quality and integrity are avialable for each planning (DQOs) and report phase (valid data).

Note: Although some of the these responsibilities may be delegatyed out, the ultimate responsibility still lies with the Project QA Official

* * The Project QA Official must be identified and documented in each QAPjP before full approval can be granted.

Element 5. Data Quality Assurance (QA Objectives)

This element is the most important section in any QA project plan. DQOs and all background information are fundamental to the development of a sound sampling design approach and of the remaining QA project plan elements.

Our office (Office of Quality Assurance) does not set DQOs, but only evaluates the appropriateness of DQOs to the sampling plan and the other elements within the QA project plan.

Purpose of DQO's

All data are subject to some error. Different types of error may be introduced at different stages of data collection. Some types of error can be controlled, while others cannot be controlled but can be recognized and described. Some types of error can be quantified while other can only be described qualitatively. The overall purpose of preparing detailed plans for data collection and quality assurance is first, to make sure that an appropriate level of control is exercised over sources of error that can be controlled (i.e., sampling variability) and second, to make sure that sufficient information is obtained to describe all known sources of error to the extent possible (i.e., old/new well design or construction).

The quality of a data set is represented in terms of five characteristics of the data: precision, accuracy, representativeness completeness, and comparability (referred to as PARCC). Brief explanations of these characteristics follow.

Precision - refers to the level of agreement among repeated measurements of the same characteristic.

Accuracy - refers to the difference between an estimate based on the data and the true value of the parameter being estimated.

Completeness - refers to the amount of data that is successfully collected with respect to that amount intended in the design.

Representativeness - refers to the degree to which the data collected accurately reflect the population, group or medium being sampled.

Comparability - refers to the similarity of data from different sources included in a single data set.

During the planning of a project or program that will involve the collection of envir onmental data, it is the responsibility of both the managers and technical personnel to define how they intend to use the data and to determine the quality of data needed to support that use.

With respect to all data collection activities the following pre-planning questions must be answered:

1) Is there a decision(s) to be made, a question(s), or some other type of problem to be solved?

2) Will the decision(s) or answer(s) depend in part on measurement data?

3) Will the data input to the decision(s) come from data based conclusions?

If the answer to the above questions are yes, then the decision(s) or question(s) should be clearly stated in order to establish the purpose for collecting data.

Also, each conclusion requiring environmental data should be clearly stated so that the specific data needed for that conclusion can be identified.

The next step in defining DQOs is to develop statements of the "universe" to which the conclusion should apply, of the level of uncertainty that will be acceptable for the conclusion, and of the amount of time and the level of resources that will be used to collect data needed for the conclusion.

The definition of the universe is needed to develop options for the sampling design. A sampling design, among other functions, defines how data collectors will identify and select the particular sites or "units" of the environment on which chemical, biological, or physical measurements will be made. Any universe may be subdivided (stratified) in different ways, and each of the subgroups may be studied to a greater or lesser degree. The choices made in in defining the sampling strata, selecting the sampling units, and allocating the number of measurements to be made for each stratum will affect the cost of collecting data and the ability to make valid conclusions about each of the strata as well as about the universe as a whole. The Program/Project Officer must have a clear definition of the universe of interest in order to design a program that will generate data that are properly representative of that universe.

The statement about the level of uncertainty associated with each conclusion will be used to determine what types and levels of error which may be tolerated in the data. No measurements system is free of error; thus, no conclusion based on measurement data can be absolutely certain. One of the central ideas behind the development of DQOs is that the level of uncertainty associated with a conclusion may be controlled through the proper design of data collection procedures and the associated QA and QC programs. By controlling the uncertainty associated with the conclusions, i.e. the components of a decision, the ultimate risk of making an incorrect decision can also be controlled.

In order to develop a design that achieves a balance between different sources of error and that controls each source of error to an appropriate level, the Program/Project Officer must investigate the anticipated effect of major sources or error on the precision and accuracy of each conclusion requiring data. These major sources include human error, error introduced by assumptions and approximations in statistical models, sampling error, and measurement error. The Program/Project Officers will need to determine how error introduced from each of these sources affects the conclusions and will need to calculate the expected precision and accuracy of each of each conclusion, taking all of the major sources of error into account. The calculations will involve assumptions about details of a sampling design being considered (e.g., total number of samples to be collected and their distribution among strata) and assumptions about the values expected in the variables to be measured. The method employed in calculating the expected precision and accuracy of each conclusion will depend on certain aspects of the data collection approach (i.e., what quantities will be measured directly and what quantities will be estimated) and on the nature of the quantity that will constitute each conclusion (e.g., mean, proprotion, percitile, slope, etc.).

The statements of time and resources will be used for making trade offs between the type and quality of data that are needed and the amount of time and money required to collect the data. Rough estimates of the time and resources limits must be known up front for the staff to develop reasonable alternatives for the decision-maker's consideration. In addition, the staff should consider as an option that the time (unconstrained) not be associated with obtaining quality information needed to make the decision.

If all of the issues just described are adequately addressed, the Program/ Project Officer's efforts will generate the following products:

- a clear understanding of each of the conclusions requiring measurement data.
- final statements of the acceptable levels of precision and accuracy associated with each of the conclusions dependent on measurement data.

° for each conclusion dependent on measurement data:

- a final definition of the population to which the conclusion is intended to apply.
- definitions of the variables to be measured.
- statements of the acceptable levels of precision and accuracy for the measurements to be made.
- a quantitative description of the effect of major sources of error (including more than measurement error) on the precision and accuracy associated with the conclusion).
- final estimates of the time and resources required to collect the data.

The final statements of the acceptable levels of precision and accuracy associated with each of the conclusiions responds to the precision and accuracy componenet of PARCC. The definition of the population associated with each conclusion addresses representativeness. The issues of "completeness" and "comparability" are included implicitly in dealing with precision, accuracy and representativeness. Missing data ("completeness") may comprise accuracy by introducing additional bias. Missing data may also comprise representativeness if there is an inordinate effect on certain of the sampling strata. On the issue of comparability, if a conclusion is expected to apply to a defined population, then the data must be comparable across that population and among any defined subpopulations (strata).

Because of the complexity of the relationship among the PARCC terms, our office's (Office of Quality Assurance) emphasis in reviewing DQOs will be to ensure that all of the necessary elements are included, and not that each of the PARCC terms be expliciyly and individually addressed.

Minimum Requirements:

° A statement of the decision (s) that depend (s) on the results of this data collection activity.

° If the data collection activitly is of an exploratory nature and not formally linked with a regulatory decision, then a clear explanation of the purpose for which environmental data are needed.

° Statements of each specific question that will be addressed in the data collection activity and the type of conclusion that is anticipated as an appropriate

answer to each question. The conclusions should depend only on measurements.

° A clear statement of the way in which each conclusion of the study will be represented, in terms of the results of staticical calculations made with the measurement data. For example:

- estimates of population parameters, such as a mean, proportion or percentile;
- estimated distributions of the variables accross the population sampled;
- estimates of dose, exposure, or environmental effects based on calculations with the data.

° Statements of the acceptable levels of precision and accuracy associated with each of the conclusions dependant on measurement data as follows:

- a statement of the acceptable amount of variance or imprecision (e.g., either confidence intervals or probabilities of incorrectly accepting or rejecting a hypothesis (Type I and Type II errors.)

- a description of any expected bias, including a statement of acceptable amount and direction of bias if this can be anticipated.

° A definition of the population to which each of the conclusions is intended to apply, including definitions of all subpopulations or strata.

° Definitions of the variables (e.g., ambient concentration of polluntant "a" in medium "b", measured in "x" units) that will be measured.

° The acceptable levels of precision and accuracy for the measurements to be made.

- for each matrix (medium) and parameter (variable), provide a table of the objectives for: a. Accuracy b. Precision c. Sentivity or method detection limits.

° A flow chart or spread sheet illustrating the relationship between the measurement data and each conclusion that will be made with the data. The chart should diagram the steps that will be needed in order to evaluate the data and draw a conclusion. The chart should also present the results of statistical analysis used to evaluate the effects of major sources of error on the precision and accuracy of each conclusion dependent on the data.

*For Laboratories.

Minimum Requirements

° For each matrix (medium) and parameter (variable) provide a table of the analytical data quality objectives for:

- Accuracy
- Precision
- Sentivity or method detection limit
- Completeness

° Other sources of error that should be dicussed, include, but are not limited, to the following:

- Laboratory Practices (See Element Number 6a)
- Outliers (they should be statistically determined)
- Reduction and validation errors.
- Internal quality control procedures.
- other

ELEMENT 6. SAMPLING PROCEDURES

Purpose: This element should succinctly describe the sampling rationale, sampling design, sampling procedures, and all other components of a project's collection activities. Inadequate planning will often lead to biased, meaningless, or unreliable results; good planning, on the other hand, can can produce valid results. The quality and utility of analytical data depends critically on the validity of the sample and the adequacy of the sampling design. The selection of the optimum sampling design is one of the most important factors influencing the reliablity of data. Please refer to Data Quality Objectives (ELEMENT 5).

Minimum Requirements for QA Plans

° Provide sufficient documentation of the sampling rationale (supported by the project desciption), sampling design, sampling procedures and other sample collection activities to enable reviewers to adequately evaluate the appropriateness of this element to the Data Quality Objectives, analytical procedures, internal quality control samples and procedures and other elements of the project or Laboratory plan (if laboratory is envolved with sampling activities).

- a succinct justification of the project sampling rationale by matrix location, strata, population, measurement parameter or any other charateristics.

- a detail description of the sampling design

  a) specifing the locations of the sampling sites
  b) number of samples to be collected per matrix
  c) collection frequency
  d) the population to be sampled (including subpopulations)
  e) defining the sampling strata
  f) other relevant factors which may influence the design of the sampling approach; i.e., homogeneity of the universe, accessibility of the sampling area, sampling conditions, well design or construction, etc.

° Provide a map showing sampling sites, strata and other relevant factors (i.e., well locations, atypical habitats, etc.).

° Provide flow diagram(s) or charts(s) delineating sampling program operations.

° Identification of sample custodian(s). (need not indentify, if identified in ELEMENT 4).

° Provide a complete description of the sampling procedures or SOP(s). These procedures should be documented in the QAPjP as an appendix.

° Provide a table detailing sample preservation methods, maximum holding times and types of containers to be used.

° Document all special conditions for preparation of sampling equipment and containers to aviod sample contamination (i.e., containers for organics should be solvent-rinsed; containers for tracemetals should be acid-rinsed; containers for bacteria should be sterilized).

- must include specific decontamination procedure(s).

° Provide examples (exhibits) of forms, notebooks and documents to be used in recording data collection activities (See ELEMENT 7).

° Provide detailed descriptions and/or criteria of Good Field or Management Practices (also see ELEMENT 6a).

- The following Good Field and/or Management Practices should be developed (** written procedures or SOPs) and implemented in all QA project and Laboratory Plans (where applicable):

**For each written procedure, the following information should be included:

1) the responsible individual(s).

2) the review and evaluation process and frequency of review

3) the quality control criteria (where applicable)

4) the filing and/or storage procedures and codes for retrieving those files (login and logout procedures).

A. Administrative procedures:

- correspondences (letters and memorandums)
- QA/QC reports
- Data reporting and checks
  - errors
  - completeness
- procurement procedures (QC criteria)

B. Documentation:

- Field activities (sample tags, chain of custody forms, notebooks, etc.).
- Procedures for filing and storages of records
- Records retention time frames (Storage)

C. Review and evaluation:

- Sampling plans (site investigation plans, project operation plans, etc).
- Sampling designs (statistical or professional judgement).
- Field construction activites (well drilling, foundations, dikes, soil liners, leachate collection systems, etc.)
- Field Standard Operating Procedures (on a annual basis)
- Field instrument and equipment quality control criteria in procurement requests.

D. Quality control procedures:

- To ensure adequate supplies and spare parts (standards, reagents, preservation material, sample containers, etc.).
- Field decontamination procedures.
- Corrective actions on equipment/procedural problems or failures.
- Standard operating procedures are implemented.

- Maximum holding times and proper sample containers.
- Field quality control samples and their frequencies.
- Field or management data validation procedures.
- Storage, packaging and shipment of samples.
- Field calibration/preventative maintenance procedures.

E. Data processing, review and reporting:

- Quality control checks on procedures and frequencies
- Computer quality control checks on inputs, outputs, and verification of softwares
  - procedures
  - frequency of checks

ELEMENT 6a: GOOD LABORATORY PRACTICES

Purpose: Laboratories inherently have activities prior to and following analysis which directly or indirectly affect the quality of data. To ensure that reliable and defensible data has been generated and that all sources of error (internally and externally) have been identified (See ELEMENT 5), every laboratory must maintain an acceptable level of Good Laboratory Practices (GLPS).

Minimum Requirements:

- Provide a general description of GLPs that have been developed and implemented in your laboratory.
- Provide a table detailing the sample preservation technique, maximum holding times and the types of containers required per parameter (variable) or parameter group.
- Document all special conditions for preparation of sampling equipment and containers to avoid sample contamination per parameter group (i.e., organics, trace metals, bacteria, radiochemical parameters).
  - Include all specific routinely used decontamination procedures.
- Provide complete Standard Operating Procedures for recording data in forms, notebooks, computers, etc. and how records are to be identified and stored. (also see ELEMENT 6).
- Provide a flow chart outlining the major laboratory activities.
- Provide detailed description's and criteria for Good Laboratory practices not addressed in other elements. See the following pages.

The following Good Laboratory and Management Practices should be developed and implemented in all QA Projects and Laboratory Plans (were applicable):

A. Administrative Procedures:

1. Records filing and storage procedures
2. Correspondance procedures (letters, memorandums, etc.)
3. QA/QC reporting procedures
4. Data reporting procedures
   - Quality control checks on errors and completeness.
5. Procurement request (quality control criteria).

B. Facility Quality Control Requirements:

1. Should include, but not limited to, the following items:

   a. ventilation
   b. compressed air
   c. humidity
   d. temperature
   e. electricity and voltage controls
   f. noise levels
   g. storage (cold room, chemicals, walkin incubators, etc.)
   h. location of microbial, chemical, radiochecical laboratory sections (i.e., the microbial and chemical lab sections must not be located in the same room/area without a physical division/partion.

2. Quality control criteria should be established for each item identified.

3. Quality control criteria should be incorporated into procurement requests.

4. Should identify the responsible individual that will ensure the quality of the items identified.

C. Equipment/Instrument Quality Control Requirements:

1. Items that should be covered include, but not limited to the following:

   a) analytical instruments/laboratory equipment.
   b) furnaces
   c) incubators
   d) generators
   e) refrigerators
   f) laboratory hoods

g) equipment/instrument parts
h) equipment/instrument services contracts
i) chemical, microbial, radiochemical and volumetric tolerance of laboratory storage containers.

2. Quality control criteria should be established for each item identified.

3. Quality control criteria should be incorporated into procurement request.

4. Should identify the responsible individual that will ensure the quality of the items identified.

D. Laboratory Material Quality Control Requirements:

1. Should include, but not limited to, the following items (for each analytical method):

a) grades of reagents
b) grades of solvents
c) grades of gases
d) grades of membrane filters
e) grades of microbial media
f) grade of distilled/deionized water

2. Quality control criteria should be established for each item identified (per analytical method).

3. Quality control criteria should be incorporated into procurement request.

4. Should identify the responsible individual that will ensure the quality of the items identified.

E. Storage Requirements for Laboratory Material:

1. Items that should be covered include, but not limited to, the following:

a) reagents, solvents, gases
b) microbial media
c) samples, standards, blanks
d) sample extracts
e) radiological materials, and samples
f) light sensitive reagents and solvents.
g) microbial cultures
h) Hazardous waste, extracts, etc.

2. Quality control criteria should be established for each item identified.

3. Quality control criteria should be incorporated into storage procurement requests (also see C.)

4. Should identify the responsible individual that will ensure the quality of the items identified.

F. Disposal of Hazardous Waste:

1. Should develop and implement disposal procedures

2. Identify and establish quality control criteria for the disposal of hazardous waste.

3. Quality control criteria should be incorporated into equipment, supplies, containers, and other procurement requests.

4. Should identify the responsible individual that will ensure proper storage of hazardous waste.

G. Data processing, review and reporting:

1. Items that should be covered include, but not limited to, the following.

a) manual data processing procedures
b) computer data processing procedures
c) data package completeness

- raw data
- calculations
- calibration graphs, charts
- strip charts
- GC/MS printouts
- method detection limit
- etc.

d) manual data package review
e) computer data inputs and outputs reviews
f) verification procedures for computer software
g) quality control checks (procedures) and frequencies for a thru f above.

- manually
- use of reference materials (for computerized instruments)
- use of more rigorous software programs.
- etc.

2. Quality control criteria should be established for data processing, reviews, and reporting.

3. Quality control criteria should be incorporated into equipment and supplies (i.e., computers, softwares, paper printing quality, etc.).

4. Should identify the responsible individual that will ensure the quality of the data processing, reviews, and reporting.

H. Glassware Cleaning Requirements

1. Items that should be covered include, but not limited to, the following:

   a) cleaning based on substances to be removed
   b) cleaning based on analytical requirements
   c) cleaning based on sampling requirements
   e) cleaning based on biological requirements

2. Quality control criteria should be established for each item identified (down to specific methods, if required).

3. Quality control criteria should be incorporated into cleaning material procurement requests.

4. Should identify the responsible individual that will ensure the quality of the items identified.

I. This section should reference the other elements in the QA plan were Good Laboratory Practices are addressed.

For example: ELEMENT 7: Sample custody
ELEMENT 8: Calibration procedures
ELEMENT 9: Analytical procedures
ELEMENT 10: Data reduction, validation and reporting
Etc.

ELEMENT 7. SAMPLE CUSTODY PROCEDURES

Purpose: Sample custody procedures are necessary to maintain and document sample possession; to adequately establish and/or support the use of sample data in potential enforcement, regulatory or legislative actions.

Our office recommends that EPA National Enforcement Investigation Center (NEIC) or equivalent sample identification, documentation and chain-of-custody procedures be used.

(NEIC Policies and Procedures, EPA-330/9-78-001-R, Revised February 1984).

The following Sample Custody should be adopted.

A sample is under custody if:

1. It is in your possession, or
2. It is in your view, after being in your possession, or
3. It was in your possession and you locked it up, or
4. It is in a designated secure area.

Minimum Requirements

Field

- Document the procedures for preservation of reagents or supplies which become an integral part of the sample.
- Document the procedures for identifying samples to be collected.
  - Prepared sample labels
- Document the procedures and forms (notebooks) for recording the exact location, analysis to be performed, sample history, sampling conditions, etc.
- Document the field custody procedures and provide examples of all forms that will be used during the project.

Laboratory

- Document the procedures for receipt of samples.
- Document the forms (notebooks) for recording (logging) samples received/transfered within the laboratory.
- Document the laboratory custody procedures and provide examples of all forms that will be used during the project.

Project Documentation

It is the responsibility of all organizations to ensure that all project documents issued to or generated by organizations will be accounted for when the project is completed. Therefore:

- Develop and implement procedures for documenting projects (Refer to NEIC Policies and Procedures, EPA-330/9-78-00-R, Revised Feb., 1984.
  - serialized document control system.
  - document inventory procedures
  - an evidentiary filing system

Sample Identification:

The method of identification of a sample depends on the type of measurement or analyses performed. When in-situ measurements are made, the data are recorded directly in logbooks or field data records, with identifying information (project code, station numbers, station location, date, time, samplers), field observations, and remarks. Examples of in-situ measurements include pH, temperature, conductivity, flow measurement, continous air monitoring, stack gas analysis and OVA.

Samples, other than in-situ measurements, are identified by a sample tag or other appropriate identification (hereafter referred to as a sample tag).

These samples are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often separated into portions depending upon the analyses to be performed. Each portion is preserved in accordance with applicable procedures and the sample container is identified by a sample tag. The information recorded on the sample tag should include the following:

Project Code

Station Number

Date

Time

Station Location

Samplers

Remarks

Preservative used

Type of analysis required

Lab Sample No. (May be completed by the receiving laboratory)

The sample tag contains an appropriate place for designating the sample as a grab or a compositeand identifying the type of sample collected for analyses. When used for air samples, the sampler may use the remarks section to designate the sequence number and identify the sample type. The Project Officer will detail procedures for completing tags used for soil, sediment, and biotic or other samples. The sample tags are attached to each sample or container.

After collection, separation, identification, and preservation, the sample is maintained under chian-of-custody procedures discussed below. if the composite or grab sample is to be split, it is aliquoted into similar sample containers. Identical information is completed on the tag attached to each split and one is marked "Split". In a similar fashion, tags will be marked for "Blank" or "Duplicate" samples.

Field logbooks are used to document all field activities and will ensure the validity of the samples collected. All information of the field activities should be recorded into a logbook. The logbook(s) should include the following information:

- Location of the sampling points
- Purpose of the sampling (i.e., defining pit areas, plumes, etc.)
- The environmental setting
- The number and amount of samples taken or required
- Weather conditions
- Field observations and measurements
- Description of sampling points
  - photographs
  - maps
- Date and time of collection(s)
- Type of preservative used
- Analysis, laboratory distribution or storage requirements
- The types and quantities of standards and/or reagents used

Field Custody Procedures

1. Collect only the number of samples neeeded to represent the media being sampled. To the extent possible, determine the quantity and types of samples and sample locations prior to the actual field work. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

3. Sample tags shall be completed for each sample, using waterproof ink unless prohibited by weather conditions. For example, a logbook notation would explain that a pencil was used to fill out the sample tag because a ball point pen would not function in freezing weather.

4. The Project Officer should determine whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment

1. Samples are accompained by a Chain-of-Custody Record. When transferring the possession of samples, the individuals relinguishing and receiving will sign, date, and note the time on the record. This Record documents sample custody transfer from the sampler, often through another person, to the analyst in a mobile laboratory or at the laboratory.

2. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment (e.g., one for each field laboratory, one for samples shipped, driven, or otherwise transported to Lab). Shipping containers will be padlocked or sealed for shipment to the laboratory. The method of shipment, courier name(s) and other pertinent information is entered in the "Remarks" section on the custody record.

3. Whatever samples are split with a source or government agency, a separate Receipt for Samples form is prepared for those samples and marked to indicate with whom the samples are being split. The person relinguishing the samples to the facility or agency should request the signature of a representative. If a representative is unavailable or refuses to sign, this is noted in the "Received by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time.

4. All shipments will be accompained by the Chain-of-Custody Record identifying its contents. The original record will accompany the shipment, and the copy should be retained by the Project Officer.

5. If sent by mail, the package will be registered with return receipt requested. Freight bills, post office receipts, and Bills of Lading will be retained as part of the permanent documentation.

Receipt for Samples Form

A completed Receipt for Samples form complies with these requirements and is used whenever splits are provided. This form must be completed and a copy given to the owner, opertor, or agent-in-charge even if the offer for split samples is declined. The original is retained for the Project Officer.

Laboratory Custody Procedures

1. A designated sample custodian accepts custody of the shipped samples and verifies that the information on the sample tags matches that on the Chain-of-Custody Records. Pertinent information as to shipment, pickup, courier, etc. is entered in the "Remarks" section. The custodian then enters the sample tag data into a bound logbook which should be arranged by project code and station number.

The laboratory custodian will use the sample tag number or assign a unique laboratory number to each sample tag and assure that all samples are transferred to the proper analyst or stored in the appropriate secure area.

2. The custodian distributes samples to the appropriate analysts. Laboratory personnel are responsible for the care and custody of samples from the time they are received until the sample is exhausted or returned to the custodian.

3. When sample analyses and necessary quality assurance checks have been completed in the field, the unused portion of the sample must be disposed of properly. All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation. Samples received by the laboratory should be retained until after analyses and quality assurance checks are completed. When investigative documents are requested, for the evidentiary file, all identifying tags are removed for retention in the permanent documentation. Sample containers and remaining sample material should be disposed of appropriately.

4. Samples of materials which have been associated with high hazard levels should received in a specialized regulated laboratory. This laboratory reduces the hazardous characteristics of these samples and prepares them for routine analysis. To avoid potential contamination, tags from samples received by the laboratory are not considered permanent documents and will not be incorporated into the evidentiary file. The laboratory will verify that the information on arriving sample tags is accurately recorded on the appropriate Chain-of-Custody Records and notify the project manager or officer of any discrepancies. The sample tag number is entered on the Chain-of-Custody Record in the "comments" column. regulated laboratory personnel will initial the entry after verifying sample tag data or resolving a descrepancy.

5. The laboratory will submit a memorandum to program officer when the project documents are assembled. The memorandum, to be retained in the evidentiary file, certifies that the sample tags have been appropriately disposed of together with the sample containers and any remaining portions.

6. Data magnetic tapes will be copied into the appropriate lab minicomputer disc files. The original tapes will then be stored in the locked cabinets and the disc data will be used for computer data processing.

ELEMENT 8. CALIBRATION PROCEDURES AND FREQUENCY

Purpose: Calibration procedures (analytical & field) and their frequencies (recalibration) serves as a quality control check on the bias of instruments during the portion of the analysis.

Minimum Requirements:

For each measurement parameter (or parameter group) the following information sholud be documented:

- Provide a written description, Standard Operating Procedure, or reference the applicable manufacture procedures (manual).
- Provide the frequency for recalibration (internally and externally).
- List the calibration standards to be used and their sources, including traceability procedures.
- Prepare a QA/QC review audit flow chart showing the organizational level and key individuals who will review the calibration procedures.
- The calibration procedures should contain, but not limited to, the following items:
  - equipment indentification number (code)
  - calibration schedule (in-house, externally)
  - any specific equipment specification that may be required
  - criteria for selecting equipment to meet any equipment specifications
  - specific step-by-step procedures
  - equipment calibration log sheet
    a) Date of calibration.
    b) All information pertain to calibration procedures (i.e., maintenance problems, equipment failures, etc.).

c) Document the individual who calibrated the instrument and

d) ensure that all adjustments have been made

e) Document all equipment failures.

f) Corrective action procedures (if instrument is out of order).

g) All information pertain to calibration procedures should be included (i.e., reocurring maintenance problems).

ELEMENT 9. ANALYTICAL PROCEDURES

For each matrix (or matrix group) and parameter the following information is required.

a) Provide a written description (SOP) of the analytical procedure or reference the applicable EPA, ASTM, or Standard Methods procedures.

b) Each analytical procedures should contain the sensitivity or method detection limit.

- This can be addressed in ELEMENT 5.

*Analytical procedures also includes geotechnicals, microbial, aquatic, biochemical, earth science methods or any other environmental measurement methods.

OFFICIALLY APPROVED OR RECOMMMENDED EPA PROCEDURES WILL BE USED WHEN AVAILABLE.

ELEMENT 10. DATA REDUCTION, VALIDATION AND REPORTING

Minimum Requirements:

For each major measurement parameter (or measurement method), describe the following items:

° Document the principal criteria that will be used to validate data integrity, at minimum it should include the following:

- Data Logging
  a) Verify all paperwork, chair-of-custody forms, etc.
  b) Verify all holding times, preservations and containers.
- Completeness of analytical data.
- Corrections of analytical data.
  a) a check on all mathematical calculations
  b) a check on all data transpositions.
  c) a check on all units of measure.
  d) a check on all significant figures.
  e) a check on all instrument's calibrations, tunings, and performances.
  f) etc.
- Accuracy
- Precision
- Representativeness

° Methods used to identify and treat outliners, all outliners should be statistically evaluated.

° Provide all equations used to calulate the concentration or value of the meassured parameters and reporting units or reference the applicable SOP or EPA, ASTM, Standard Methods procedures [If an SOP is referenced, (other than EPA, ASTM or Standard Method) then the SOP must be appendixed.]

° Provide a data flow chart from collection of raw data through storage of validated concentrations with the organization level and key individuals who will review or handle the data.

° Provide the reporting and the QA/QC review procedures (internally and externally).

ELEMENT 11. INTERNAL QUALITY CONTROL CHECKS.

Purpose: Internal and external Quality Control Check samples and procedures are used to provide a measure of the consistency of samples and to provide an estimate of variance and the bias in the collection process, handling processes (such as sample shipping, storage, and preparation), and analyses.

Other quality control checks that should be documented or referenced such as, construction and review of quality control charts (Shewhart or Cusum chart); calibration procedures; preventive maintenance procedures; data reduction/validation procedures; quality control check sample programs; performance evaluation studies; the traceability of instrument standards, samples and data; analytical and QC methods, sample preservation and transportation procedures; and audits.

Minimum Requirements:

- Identify and briefly describe each quality control check sample and procedures that is or will be incorporated into the project and that will meet the Data Quality Objectives of the project or Laboratory.
- For each quality control check sample and procedure document the frequency of use or review. Our office recommends that QC samples be analyzed at a 10% frequency.
- Provide a flow chart showing intergration of the quality control check samples, procedures and review procedures.

The table 11-1 presents the breakdown of QC samples used in previous projects (studies).

Table 11-1. Quality Control Check samples

Sample	Comments
Field Blanks	Analyzed to detect accidental or incidental contaminations.
Sample Bank Blanks (Method Blanks)	A field blank passed through the sample preparation and operators, after cleaning, to check for residual contamination.
Contamination Blanks	A field blank passed through equipment and/or samples to check for residual contamination.
Reagent Blank	A blank to check reagent contamination level.
Calibration Check Standard	A standard for extract matrix effects on recovery of known added analyte.
Spiked Sample (Field Matrix Spike)	To check for sample and extract matrix effects on recovery of known added analyte.
Total recoverable	A split sample (a second aliqueot) is digested by a more vigorous method to check the efficiency of the protocol method.

Sample	Comments
Split-Extract (Lab split)	To check sample, injection and instrument reproducibility.
Duplicate Sample	To determine total random error.
Triplicate Sample	The prepared sample is split into three portions to provide blind duplicates for the analytical laboratory and a third replicate for a referee laboratory to determine interlab precision.
Internal Standards (Spikes)	An analyte which mimics the behavior of target analytes and is added to extract prior to analysis, to check on instrument performance.
Surrogate Sample	An analyte which mimics the behavior of target analytes, and is added to field sample or lab extract, to check for sample/extract or extract matrix effects on recovery of known added analyte.
Indicator Sample	Usually a qualitative or semi-qualitative parameter (method) used to indicate the presents of specific analytes.

ELEMENT 12. MANAGEMENT, DATA QUALITY, TECHNICAL SYSTEM AND PERFORMANCE AUDITS

Purpose: Project audits provide assurance that the quality control job is being done effectively. Audits will serve to:

- Provide to management an on-going assessment of the quality of the results produced by the organizations data collection activities and how well data quality objectives (DQOs) are being met.
- Identify areas where improvement in the QA will result in increased reliability of data.
- Ensure that the QA program as defined by the QA Project Plan is implemented.
- Demonstrate that a organization is actively assessing the effectiveness of its QA program.
- Evaluate appropriateness of resource levels applied to QA.
- Provide a measure of the organization's commitment to effective corrective action when audits identify areas of concern.
- Provide suggestions for alternative ways of accomplishing QA tasks or dealing with QA problems.

Below are the four basic audits that each project (or laboratory) plan should describe (both in-house and extramurally). Some of these audits may be an ongoing process (Management), crossing over several projects, but affects each project and thus should be documented in each QA project plan.

Management Audits

Management audit is a systematic investigation to determine whether management functions and responsibilities related to environmental measurements are performed in accordance with appropriate quality assurance guidance. They are a review of the implementation of the approved QA plans. They evaluate the QA program of an organization responsible for environmental data collection activity in all its dimensions:

- The level of financial resources and personnel devoted to implementing the QA program.
- The level of management support.
- Tracking systems.

- Criteria for classifying data collection projects, according to how stringent the QA need to be and how extensive the documentation needs to be.

- Procedures for developing DQOs.

- Procedures for developing and approving QA Project Plans (QAPjPs).

- The Quality of existing QAPjPs.

- Procedures for developing and approving Standard Operating Procedures (SOPs).

- Procedures and schedules for conducting audits.

Data Quality Audits

Data quality audit is a systematic investigation to determine whether data derived from an environmentally realted measurement is of known quality. A data quality audit focuses on collected data and it will determine whether or not sufficient information exists with the data set to support an assessment of data quality. Data quality audits evaluates:

- A data set, or all the data sets of a particular project, against its data quality objectives (DQOs).

- Whether or not the organization collecting or using the data, performed its own date quality assessment, and

- Heeded the results of its assessment in terms of whether or not the dat could be used to support its decesion.

- Whether or not an organization identified deficiences (if they existed) and corrected the causes(s), both technical and managerial.

Technical Systems Audits (Field and Laboratory Audits)

Technical systems audit is a systematic investigation to determine whether data collection and analytical technologies are sufficient to meet the data quality objectives. Technical system audits evaluates:

- Field and analytical measurement procedures (SOPs).

- Field and laboratory chain of custody procedures and records.

- Internal quality control procedures.

- Control charts.

- Field and laboratory calibration procedures and records.
- Maintenance procedures and repair records.
- Field and laboratory corrective action procedures.
- Validation, reduction and reporting procedures.
- Equipment and facilities (field and laboratory).
- Support systems (field & laboratory).
- General laboratory cleanliness.
- Other

Performance Evaluation Audit

Performance evaluation is the means of evaluating the performance of laboratory technician and the instruction or analytical systems on which they work. A PE audit is accomplished by providing PE samples containing specific pollutants (in the appropriate matrix) unknown to the technician in their identity and/or concentration. Performance evaluations are implemented externally by the EPA Office of Quality Assurance, EPA Project Officers or laboratory management and enternally by the organization's QA Offical or Project Officer. Some National Program Offices, notably the National Pollution Discharge Elimination System (NPDES) and the Office of Drinking Water programs have annual nation-wide PE audits.

Minimum Requirements:

- Develop written procedures (SOPs) for audits. If audits have not been developed, a schedule for developing audits must be included.
- Describe how the audits will be intergrated and implemented [internally (routinely) and externally].
- Identify and describe all audits planned for the project or laboratory Include any current or recent EPA audits (i.e., PE Studies, laboratory audits within the last year).
- Document any in-house audits that may affect or be intergrated with specific project audits.

- Field and laboratory calibration procedures and records.
- Maintenance procedures and repair records.
- Field and laboratory corrective action procedures.
- Validation, reduction and reporting procedures.
- Equipment and facilities (field and laboratory).
- Support systems (field & laboratory).
- General laboratory cleanliness.
- Other

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° Identify and describe all audits planned for the project or laboratory Include any current or recent EPA audits (i.e., PE Studies, laboratory audits within the last year).

° Document any in-house audits that may affect or be intergrated with specific project audits.

ELEMENT 13. PREVENTIVE MANITENANCE PROCEDURES.

Purpose: To insure that all facilities equipment (including field equipment) service's instruments and any other ancillary items that are available, are properly functioning and maintained.

Minimum Requirements:

° A description of how the responsible organization(s) monitors and controls facilities equipment, services instruments and any other ancillary items (Management SOPs).

° Describe what preventive maintenance will be covered, for example laboratory instruments, field instruments, water distillation or deionization unit, glassware washing machines, incubators, etc.

° What is the frequency for inspecting equipment, instruments and any other ancillary items (in-house and by certified inspectors).

° For each piece of equipment and instrument that has the potential to significantly altering data results (i.e., D.O. probe) or has the potential for significantly altering the allocation of resources (i.e., drilling apparatus) include a list of critical space parts that should be on hand to minimize downtime.

° Preventive maintenance procedures should contain, but not limited to, the following items (per instrument/equipment):

- specific step-by-step procedures.
- maintenance log sheets and/or schedules (in-house and externally by certified inspectors).
- due dates (if applicable) for maintenance.
- document the individual(s) responsible for ensuring maintenance has been made.
- document all maintenance performed, including dates of maintenances.
- document the corrective action procedures for preventive maintenance procedures which have not been followed, and the annual review procedures of the preventive maintenance procedures.

ELEMENT 14. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY OBJECTIVES

Purpose: Data assessments are systematic procedures used for reviewing data set(s) against a set(s) of established criteria (Data Quality Objectives) to assure that the data meets the project goals. Please refer back to ELEMENT 5: Data Quality Objectives.

Minimum Requirements:

- Develop and implement data assessment procedures (program and laboratory office procedures).
- Provide a flow chart showing each phase of the data assessment review, including the mechanism for review of the data assessment procedures (network), the organizational level and the key individuals who will assess data and/or review procedures.
- Document all statistics to be used in the calculation of:

    1. Precision
    2. Accuracy
    3. Completeness
    4. Method detection limit

- Document the statistical procedures that will be employed to assess Data Quality Objectives (including confidence levels):

    Examples:
    1. Linear regression
    2. Analysis of Variance (ANOVA)
    3. Test of significances
    4. t-test for outliers
    5. Nonparametric tests
    6. etc.

ELEMENT 15. CORRECTIVE ACTION PROCEDURES

Purpose: To provide written requirements establishing and maintaining QA reporting or feedback channels to the approriate management authority to ensure that early and effective corrective action(s) can be taken when data falls below required limits. Each QA project plan shall describe the mechanism(s) to be used when corrective actions are necessary.

Corrective action should relate to the overall QA management scheme; who is responsible for taking corrective actions; when are corrective actions to be taken; who ensures that corrective actions are taken to produce the desired results, and what steps will be taken should corrective action not take place.

Minimum Requirements:

° Each measurement system must have predetermined limits to identify when corrective action is required, before data becomes unacceptable. Should include, but not limited to, the following items:

- Field equipment/procedural problems or failures.
- Laboratory equipment/procedural problems or failures.
- Control chart nonconformances.
- Broken or Lost Samples.
- Holding Times problems or failures.
- Calibration and Standardization problems and failures.
- Preventive and remedial maintenance problems.
- Sample custody and handling problems or failures.
- Sample transportation problems
- Documentation deficiencies or problems.
- etc.

- Identify the organizational level(s) and the key individual(s) responsible for initiating corrective action(s) and for approving corrective action(s).

- The Project QA Official must be notified of any major corrective action that results in a change in procedures or a loss of data. All nonconformances should be documented and reported internally (in-house) and in the final (annual) QA project report (See ELEMENT 16).

    - Therefore, the QA Project or Laboratory Plan should include procedures for documenting and reporting nonconformances.

ELEMENT 16. QA REPORTS

Purpose: The purpose of reports (communications) is to ensure that staff personnel (internally and externally) in the program offices can effectively develop and implement projects, perform activities, and resolve problems.

Minimum Requirements:

Internally

° Describe the internal mechanisms, SOPs, and reviews that are or will be performed on the measurement systems and data quality. These reports should include at a minimum:

- Periodic assessments of data quality objectives.
- Results of audits.
- Significant QA problems, corrective actions and recommended solutions.
- The level and individuals responsible for preparing the periodic reports (field, lab and management).

Externally

° Submit QA reports to the EPA Region VI Office of Quality Assurance (see below). The responsible individual for preparing this report should be the Project QA Official.

The Region VI Office of Quality Assurance will be tracking projects involving environmentally related measurements. One-time projects of 12 months duration or less, will require only a final QA report. Projects of longer duration, such as continuing multi-year programs, will require periodic QA reports to document implementation of the QA Project Plan. For example, continuous monitoring activities should be covered in an annual report summarizing the status of such projects for each annual budget period. The QA report on each project should be a separately identified Status Report containing:

A. QA management (any changes)

B. Status of completion of the QA project plan

C. Measures of data quality from the project

D. Significasnt quality problems, quality accomplishments, and status of corrective actions

E. Results of QA Performance audits

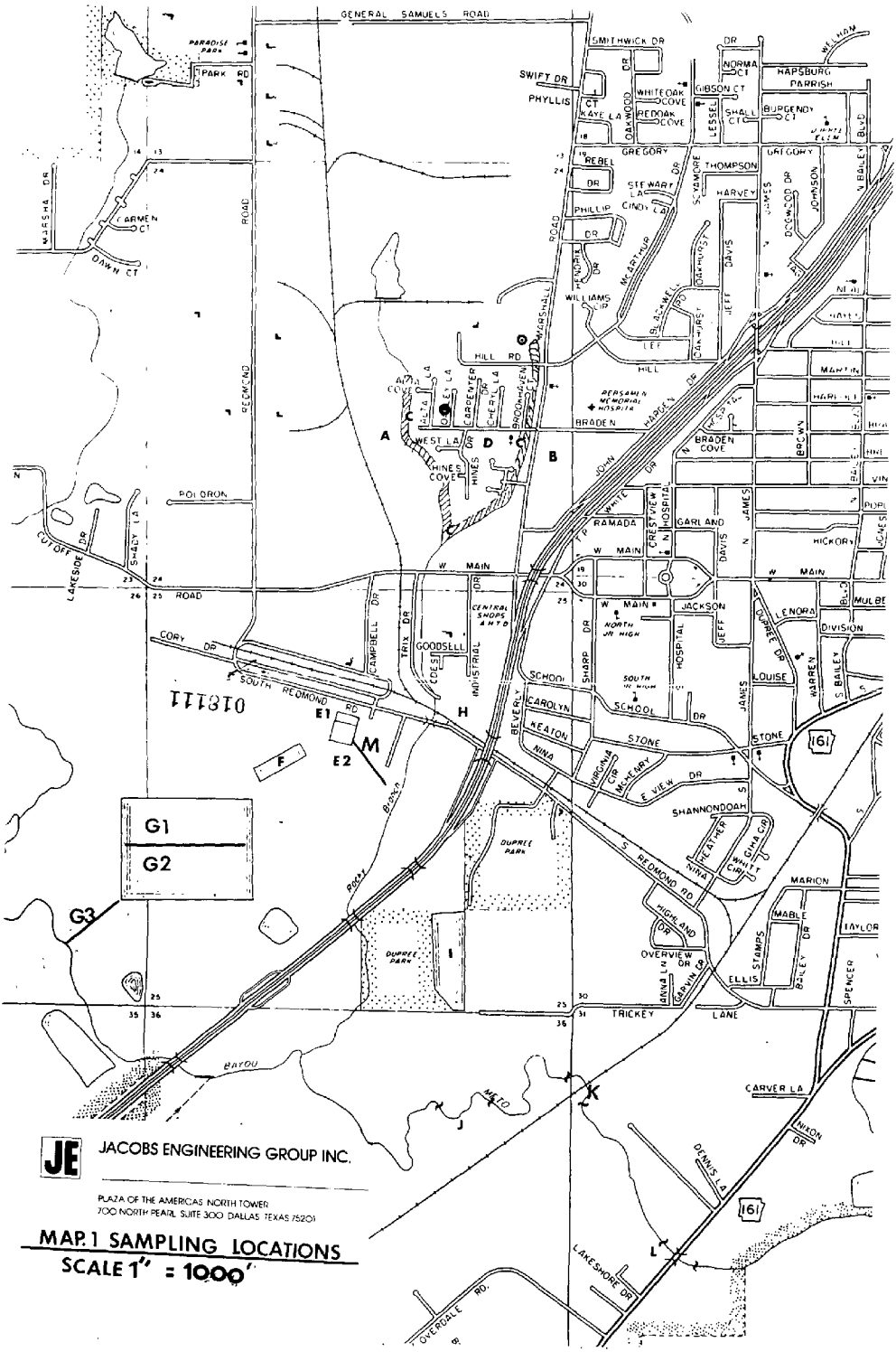
F. Results of QA Technical Systems audits

G. Results of QA Management and Data Quality audits

H. Assessment of data quality in terms of precision, accuracy, completeness, representativeness, and comparability

I. Quality Assurance related training

J. Assessment of indicators used in the project (when applicable)



JACOBS ENGINEERING GROUP INC.

PLAZA OF THE AMERICAS NORTH TOWER
700 NORTH PEARL SUITE 300 DALLAS TEXAS 75201

MAP 1 SAMPLING LOCATIONS

SCALE 1" = 1000'

018112

JE

JACOBS ENGINEERING GROUP INC.

FIGURE E

PLAZA OF THE AMERICAS, NORTH TOWER
700 NORTH PEARL, SUITE 300, DALLAS, TEXAS 75201

SAMPLE LOCATIONS
E 1 & E2
SCALE 1" ≈ 100'

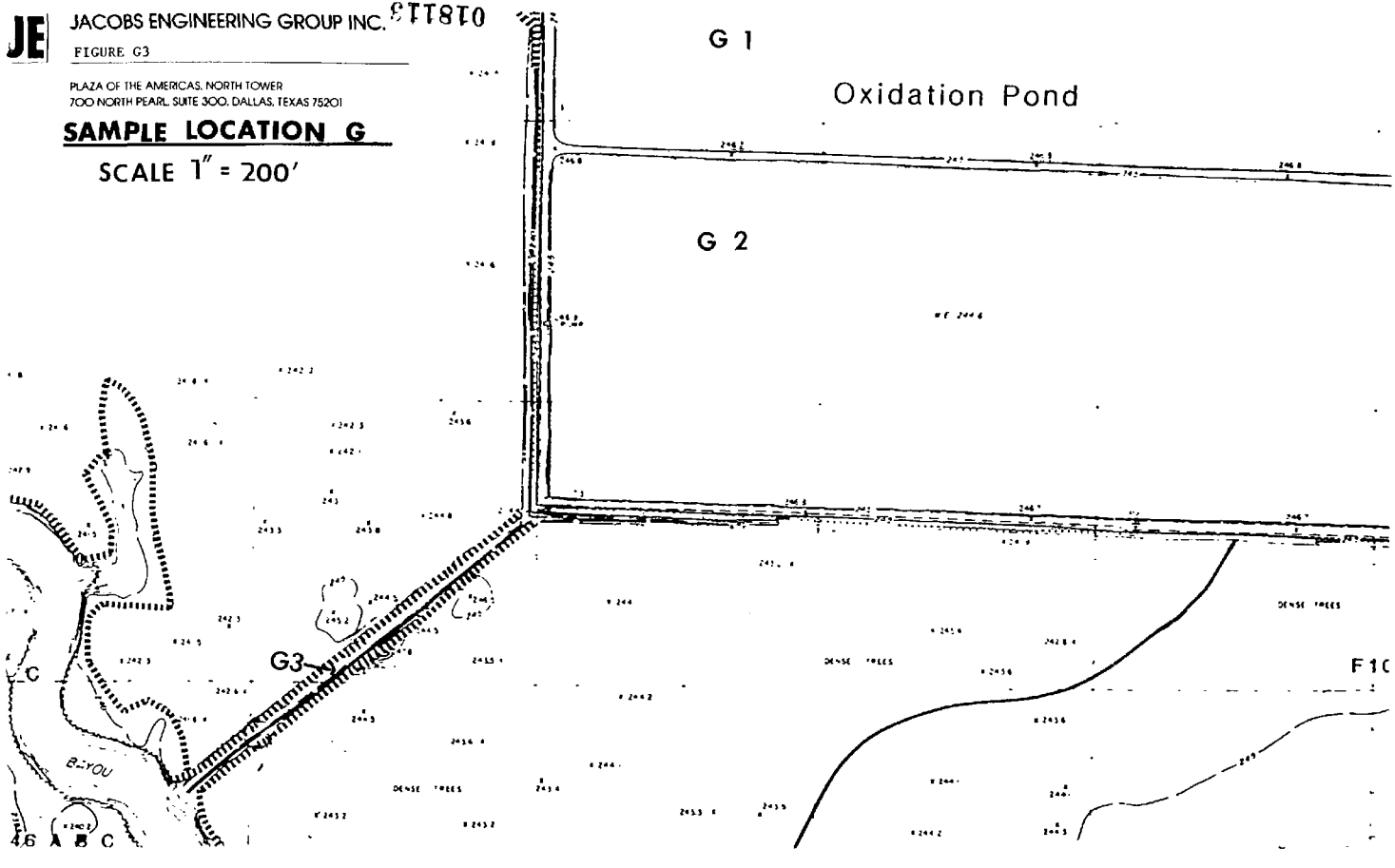
JE JACOBS ENGINEERING GROUP INC. 018110

FIGURE G3

PLAZA OF THE AMERICAS, NORTH TOWER
700 NORTH PEARL, SUITE 300, DALLAS, TEXAS 75201

SAMPLE LOCATION G

SCALE 1" = 200'

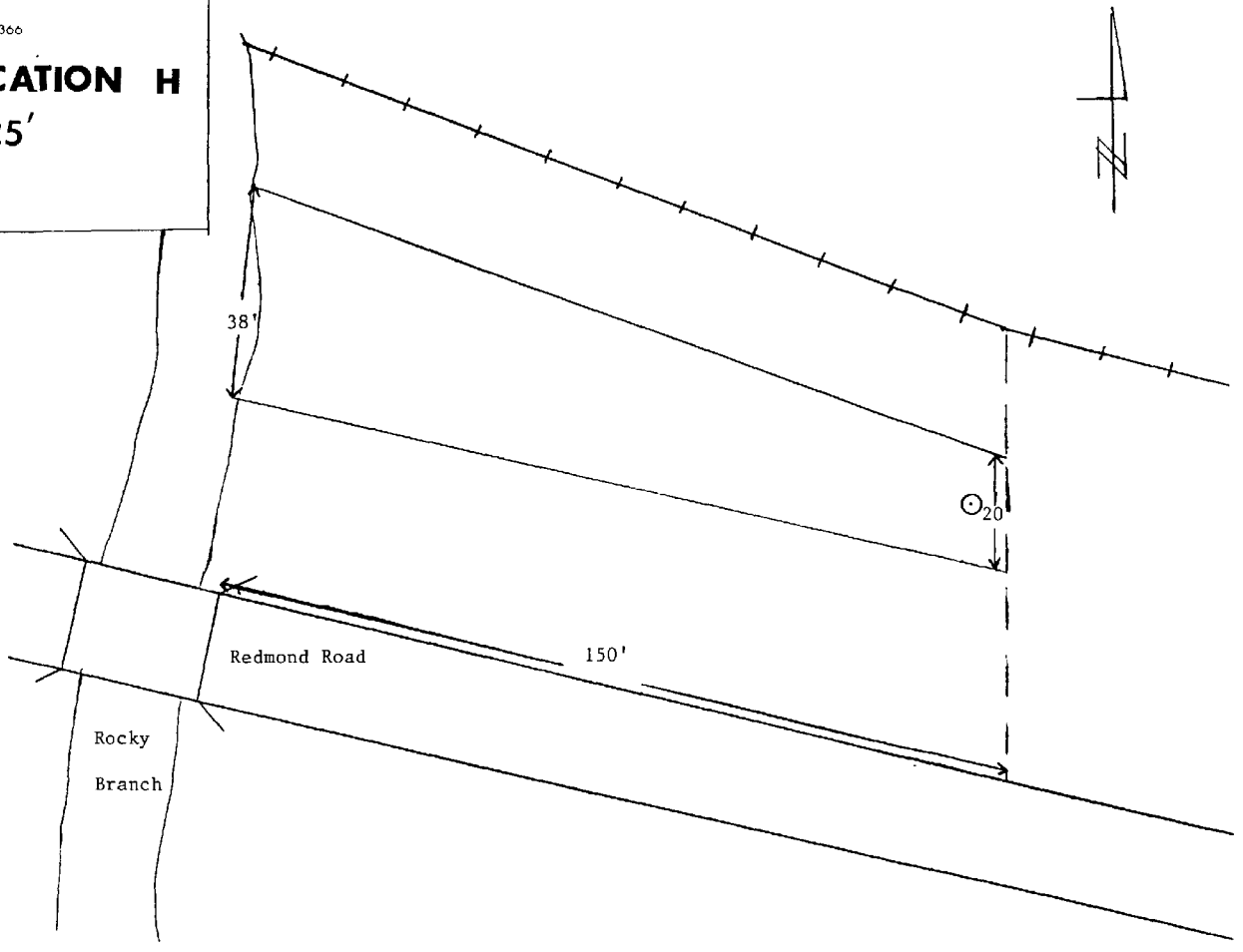


018113

JACOBS ENGINEERING GROUP INC.

700 NORTH PEARL STREET, NORTH TOWER, SUITE 300
DALLAS, TEXAS 75201
TELEPHONE (214) 969 9366

AMPLE LOCATION H
CALE 1"≈25'



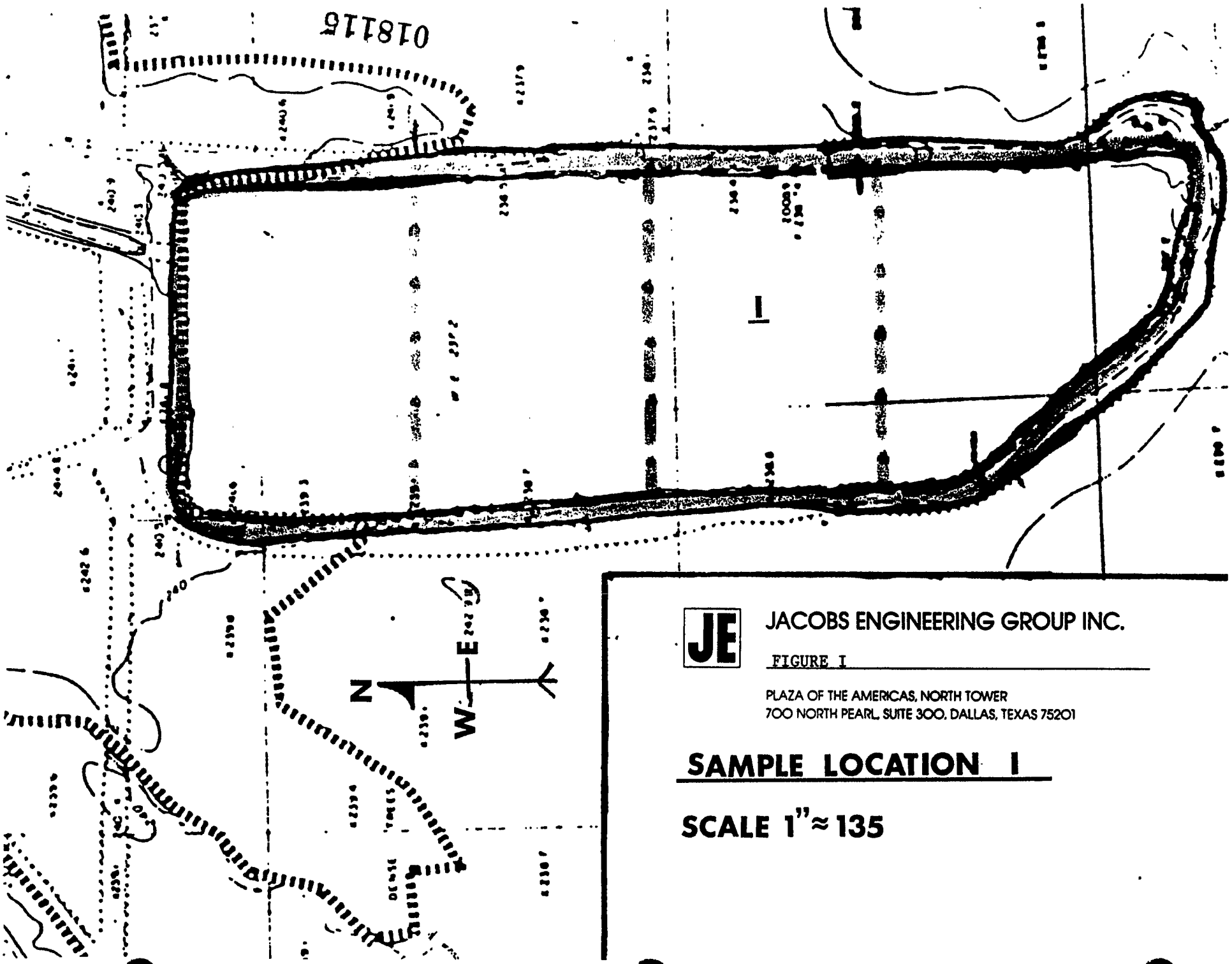


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FIGURE I

PLAZA OF THE AMERICAS, NORTH TOWER
700 NORTH PEARL, SUITE 300, DALLAS, TEXAS 75201

SAMPLE LOCATION I

SCALE 1" ≈ 135

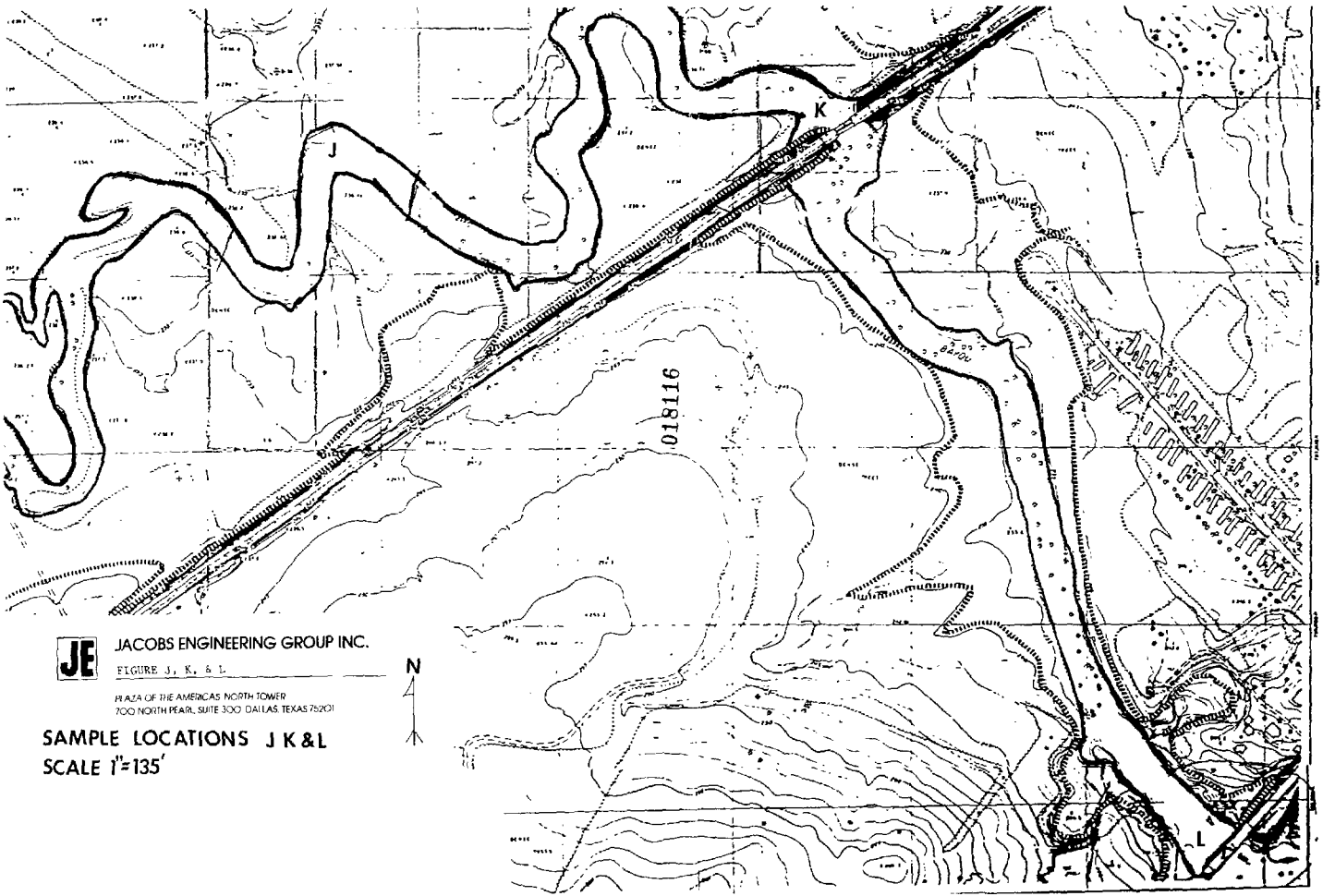

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FIGURE J, K, & L

PLAZA OF THE AMERICAS NORTH TOWER
700 NORTH PEARL, SUITE 300 DALLAS, TEXAS 75201

SAMPLE LOCATIONS J K&L
SCALE 1"=135'

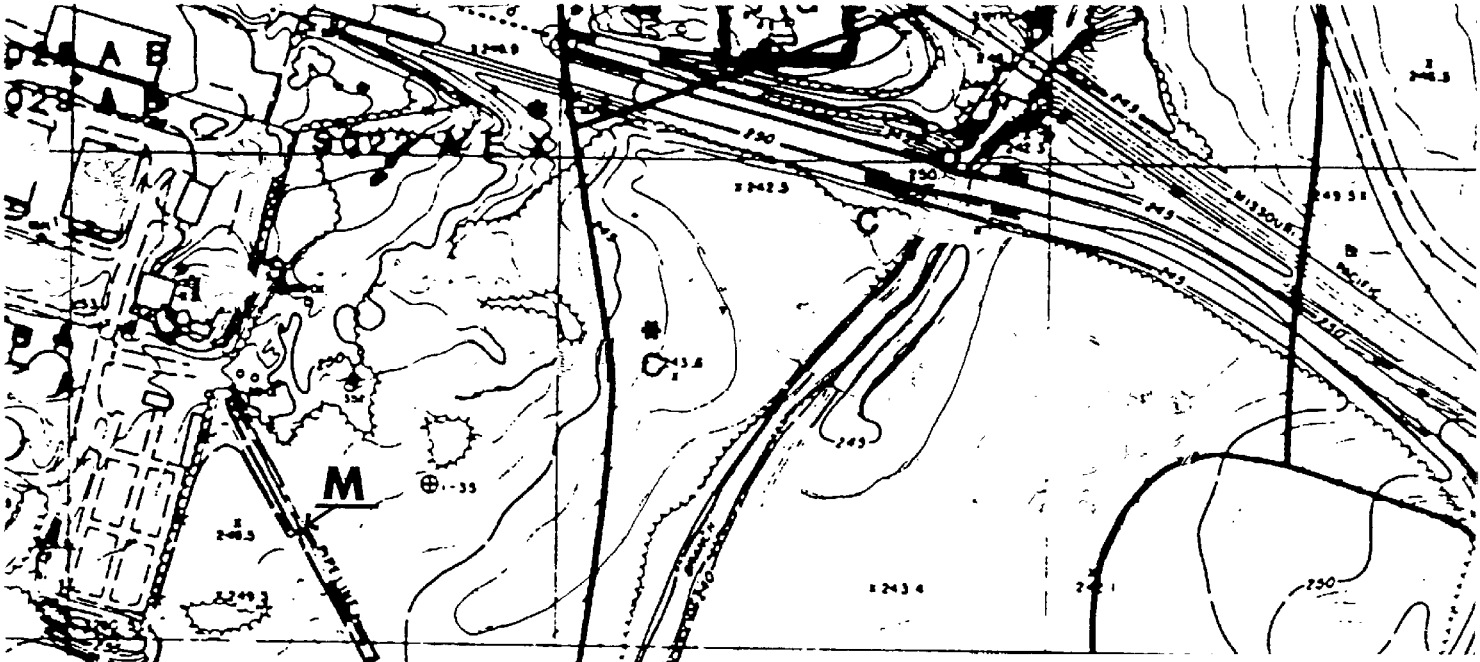

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FIGURE M

PLAZA OF THE AMERICAS, NORTH TOWER
700 NORTH PEARL, SUITE 300, DALLAS, TEXAS 75201

SAMPLE LOCATION M

SCALE 1"= 200'

018117